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DEVELOPMENT OF FLAME-RESISTANT, HIGH EFFICIENCY THERMAL INSULATION

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13. ABSTRACT *(Maximum 200 words)*

The purpose of the program was to build upon an understanding of polymeric-fiber-based thermal insulation, gained through two previous AI Research Co./U.S. Army Natick RD&E Center contracts, to develop two new flame-resistant insulator configurations. They were to be lofty, water-repellent, durable and highly efficient on a thermal resistance/weight basis. One was to be based upon staple (cut) fiber, the other on continuous filament (endless fiber). Ten inherently flame-resistant fiber candidates that were predicted to be feasible bases for highly efficient insulation were selected for laboratory trials. Initial work with them showed that flame resistance would be the most difficult-to-attain performance objective. A low insulator density target and the need to include binder material to provide insulator durability exacerbated the flammability problem. An empirical approach that relied heavily upon laboratory-scale batt making trials and flammability testing, but also included many other types of tests, eventually lead to adoption of a satisfactory, three-component, bonded, staple-fiber insulator prototype. It met all program performance objectives. Continuous filament insulator development was hindered by the lack of an adequately flame resistant, continuous filament material, by the lack of a satisfactory bonding system, and by the need to conduct many experiments on production, rather than laboratory scale.

Further pursuit of the flame resistant, continuous filament insulator approach is not recommended unless new materials become available. However, it appears that the performance, versatility and reasonable cost of the flame resistant, staple-based insulator prototype will diminish the need for an alternative configuration.

14. SUBJECT TERMS
THERMAL INSULATION, FLAME RESISTANCE, FLAMMABILITY TESTS, COLD WEATHER CLOTHING SYSTEM, CONTINUOUS FILAMENTS, STAPLE FIBERS, HIGH EFFICIENCY, FIBERS, SLEEPING BAGS, BATTING (MATERIALS), THERMAL INSULATORS

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PREFACE

This report was prepared by Albany International Research Co. under U.S. Government Contract No. DAAK60-92-C-0035. The study was conducted between May 1992 and May 1994. Ms. Judith L. Uthoff of the Natick Acquisition Center, U.S. Army Natick Research, Development and Engineering Center (Natick) was the Contracting Officer. Ms. Margaret A. Auerbach of the Textile Research and Engineering Division, Survivability Directorate, at Natick was the Contracting Officer's Representative and, in that capacity, provided technical guidance throughout the program.

Many Albany International Research Co. staff members contributed to the success of the study. These include: Ms. Lisa J. Cohen, Mr. Robert J. Coskren, Ms. Angela B. Dixon, Mr. James G. Donovan, Ms. Cynthia L. Egan, Mr. John J. Farrell, Dr. Maryann C. Kenney, Dr. Charles E. Kramer, Ms. Eda C. Kreider, Ms. Caren K. Pomar and Mr. Roman Rondiak. Ms. Cohen had primary responsibility for many of the laboratory trials and tests, Mr. Donovan was the Program Manager and supervised the day-to-day activities of the program, Dr. Kenney held overall responsibility for the contract and advised on technical and administrative issues, and Mr. Rondiak oversaw contractual matters.

Several members of Albany International Corporation's Primaloft venture staff provided assistance in planning and executing pilot trials for the staple-based, FR insulator prototype. Mr. Alan L. Billings and Mr. Robert Yando were particularly important contributors to the success of the trials.

Hoechst Celanese Corporation of Charlotte, NC participated as a subcontractor and had primary responsibility for continuous filament, FR insulator development. Reliance Products Co. of Oakland, CA also assisted in the continuous filament portion of the study. Among Hoechst Celanese staff members who made essential contributions were: Messrs. Robert B. Averell, Kevin Campbell, and H. M. (Joe) Nguyen, and Ms. Elizabeth R. Van Amerongen. Mr. Averell held administrative and technical responsibility for the subcontract and Mr. Campbell managed Hoechst Celanese' effort on a daily basis.

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UNITS OF MEASUREMENT

Almost all measurements made in the course of this work were obtained in English units. This was also the case in the two previous AI Research and U.S. Army Natick Research, Development and Engineering Center (Natick) contractual studies in the field of textile-based thermal insulation. As a result of these and other collaborative efforts, AI Research and Natick share a large body of data, in the English system, to which the results of this program will inevitably be compared. Consequently, the primary units system chosen for this report is the English System. However, all data reported in tables and graphs will be given in both the English System and the International System of Units (SI). In relatively simple tables and graphs, data will be shown directly in both systems. In more complex instances, the table or graph will be duplicated, the second one being in SI units. In these cases, the table or figure numbers will be duplicated, but given differentiation with an E or SI suffix. After careful consideration, it was decided that only English units would be used in the body of the text. Many compound units have been used in the work, that for thermal conductivity, Btu-in/hr-ft^2-°F, being an apt example. Continually repeating both sets of such compound units in the text, with the second set in parentheses, is cumbersome at best, potentially distracting, and confusing when parentheses are used for other purposes. For the sake of clarity, ideas will be expressed using a single system of units and data tables and graphs, showing values in both English and SI systems, will be referenced frequently. A set of English System to SI conversion factors will be provided in the Appendix.

DEVELOPMENT OF
FLAME-RESISTANT, HIGH EFFICIENCY THERMAL INSULATION

1. INTRODUCTION AND SUMMARY

The development, pilot line production and laboratory evaluation of two distinctly different thermal insulators was the purpose of the subject program. Although the insulators were to be alike in terms of performance characteristics, most notably flame resistance and insulating efficiency, they were to differ greatly in configuration. One was to be made of staple fiber (short, cut fiber; typically about 1.5 inches long) and the other of continuous filament (fiber that is virtually endless). Both insulators were intended for use in military cold-weather clothing systems, sleeping bags and portable shelter applications; differences between the two would ultimately influence selection for specific applications.

Imparting the desired degree of flame resistance to the insulators was foreseen as the most challenging objective. The other primary performance objective, high thermal insulating efficiency, had been an important subject in two previous U.S. Army Natick Research, Development and Engineering Center (Natick) studies performed by Al Research Co.[1,2,3] and it was anticipated that knowledge gained in those efforts would frequently be applicable. The program plan consisted of four major steps:

1. Combine our recent findings regarding insulator performance factors, as referenced above, with knowledge of state-of-the-art, inherently flame-resistant fibers to establish an efficient experimental plan for development of two flame-resistant, high efficiency thermal insulators.

2. Execute the experimental plan, which consisted of a reiterative series of fiber blending, batt forming, batt property measurement, and analysis steps.

3. Reconcile the experimental results with ten specific insulator performance targets and several other, less-well defined, but equally important insulator requirements and make choices to provide the optimal staple and continuous filament insulator configurations. This objective was frequently addressed concurrently with the experimental plan and was also, in essence, the objective of the final step in the experimental plan.

4. Produce 100 yd^2 of each of the two optimal insulator candidates for delivery to the U.S. Government and confirm, through laboratory measurement, the merit of each candidate.

The ten quantitative performance targets and the further qualitative goals mentioned in 3., above, prescribed insulator candidates that would be similar in performance and utility to Primaloft^{TM}, the product fostered by AI Research Co. development efforts, both Government and Corporate sponsored, in the 1980s. However, in addition to Primaloft-like characteristics, the new insulator candidates would have a high degree of flame resistance, which would be measured by the vertical flammability test of Federal Method 5903 and by the burning pill test of Federal Method 5907. Thus, the two insulators to be developed would:

1. Be highly efficient thermal insulators, particularly on a weight basis,

2. Be flame-resistant,

3. Have down-like compressional and recovery properties,

4. Have excellent resistance to wetting and to loss of loft when wet,

5. Be durable, especially through exposure to military laundering, and

6. Be producible at reasonable cost.

These insulator objectives, considered together, were the basis for selection of ten inherently flame-resistant (FR) fiber candidates and several bicomponent binder fiber candidates for evaluation at the outset of the program. All were obtained in staple form to facilitate mixing and batt forming trials in our laboratory. The results of these trials were, in theory, equally applicable for both staple and continuous filament selection, although the selection process for continuous filament was ultimately limited by the non-availability of most polymer types in continuous filament form. Initial fiber-material screening was accomplished through flammability testing of small batt samples, made to an appropriate standard of thickness and density. Evaluation of the flame resistance of candidate fibers and of plausible blends of candidate fibers was of first priority. Our recent past experience with fibrous insulation provided confidence that we could predict and/or manage the thermal and mechanical characteristics of batting, but the effect of each fiber candidate on flame resistance needed to be better understood and factored into the experimental plan.

All fiber and fiber blend candidates were tested for flame resistance in batts that also contained bicomponent (high melt/low melt), all-polyester, binder fiber (18%, by weight). Recent successes in

designing lightweight, durable, high performance, staple-based insulators using this relatively new type of binder[3,4] and the thermal and mechanical insulator objectives of the program made its use an almost inevitable choice for the staple-based insulator. Prospects seemed good for adapting the technology to continuous filament batting as well. Initial testing showed that the polyester binder component had a decidedly negative effect on the flame resistance of many, but not all, prospective FR fiber and fiber blend candidates. Early testing also revealed that FR polyester fiber, which had been envisaged as a technically satisfactory, cost-reducing blend component, had a similarly negative effect on the flammability of most blends. The low test density of the batt samples, 0.5 lb/ft^3, as dictated by the lightweight insulator goal, undoubtably contributed to the inadequate flame resistance measured for many seemingly credible candidates (in a 0.5 lb/ft^3 batt, approximately 99.5% of the volume is occupied by combustion-supporting air).

Development directed specifically toward the staple-based insulator approach consisted of further blending, batt-making and testing steps and a range of properties relating to insulating efficiency, compressional characteristics, water repellency and durability were addressed. However, flame resistance remained as the dominant, most difficult consideration. Flammability test results proved to be extremely sensitive to small blend ratio changes made to adjust other insulator characteristics, to changes in water repellent finish, and to binder fiber changes made in the interest of improving durability. However, one inherently FR staple fiber candidate, P84 polyimide, manufactured by Lenzing, consistently exhibited acceptable flame resistance without sensitivity to important insulator variables. P84, unlike most FR fiber, is available with producer-applied water repellent finish and in two especially useful fiber sizes, 0.55 and 1.5 denier. The lesser denier, 0.55, is unique among commercially available, inherently FR fibers, making it an essential element in terms of optimal insulating efficiency. The larger diameter fiber, 1.5 denier, is small enough to have value as an insulating fiber, and its lesser cost makes it an attractive blend component. The final staple fiber blend candidate, arrived at after a lengthy, reiterative, elimination process, consisted of:

- 0.55 denier P84 polyimide with water repellent finish (22%),
- 1.5 denier P84 polyimide with water repellent finish (60%), and
- 4 denier, bicomponent, polyester binder fiber, Hoechst Celanese Type K54 (18%).

Upon identification of this optimal staple blend, work moved from our Mansfield, Massachusetts laboratories to Albany International's Primaloft production line in Albany, New York. This line was

assembled in 1988 to manufacture blended, thermally-bonded microfiber insulation, making it especially well-suited to the needs of the program. The optimal laboratory blend was processed on the Albany line with little difficulty and the material produced met, without exception, all FR insulator performance targets established by Natick Center for the program. A relatively large experimental quantity (more than 100 yd^2) of the prototype staple insulator has been delivered to Natick Center.

Hoechst Celanese Corporation, AI's sub-contractor, assumed primary responsibility for development of the continuous filament insulator. Our role was to share in planning experiments, assist in development tasks, make all laboratory measurements on interim and final samples, and interpret and apply the results. This partnership was effective in addressing some major challenges and a seemingly endless series of technical details, but a satisfactory, continuous filament, insulator candidate could not be produced. Many obstacles contributed to this outcome and they will be reported in detail to assist in assessing the long-term viability of the FR, continuous filament, insulator concept. The underlying difficulty was the fact that important experimental steps could only be performed on a production scale; this is an inherent continuous filament issue. Scheduling, logistics, direct costs and costs due to lost production were constant complications that were compounded by technical demands for repeated experiments.

2. EVALUATION OF FLAME-RESISTANT FIBER CANDIDATES FOR USE IN STAPLE AND CONTINUOUS FILAMENT INSULATORS

A. Introduction

The work plan adopted for this development effort was a response to concern for a potentially vast, unmanageable experimental matrix envisioned after initial review of program objectives. The number of performance properties of interest, the large number of FR fibers available and the virtually limitless numbers of possible blend combinations and ratios suggested this formidable experimental matrix. However, we intended to reduce it to a tractable series of experiments by: (1) utilizing fiber manufacturers' data, (2) applying knowledge gained through recent, related work for U.S. Army Natick Research, Development and Engineering Center (Natick) and for Albany International's Primaloft Venture, and (3) striving to solve the most difficult, fundamental program objectives first, through selective experimental trials, with confidence that lesser program objectives could be addressed later through "fine-tuning" fiber blends and batt configurations.

B. Fiber Candidates

In accord with the above, ten primary, FR fiber candidates were selected for potential use in both staple and continuous filament insulators. These fiber candidates are listed in Table 1, which also reports fiber cost, effective fiber diameter, and observations regarding cost, diameter and other factors essential to the selection of each candidate. Although many fiber qualities were considered, the most important selection factors were: (1) anticipated flammability resistance, (2) fiber diameter and (3) cost. The need for flammability resistance and reasonable cost are self-evident, but the significance of fiber diameter warrants brief explanation. Previous work has shown that, within the diameter range of available FR fibers, the thermal conductivity of low density, batt-like assemblies increases as constituent fiber diameter increases[1,5,6] This is illustrated in Figure 1, which is a plot of data from earlier work[1] showing thermal conductivity as a function of fiber diameter. This diagram was used as reference during the fiber selection process. It illustrates that the program's thermal conductivity target value of $\leq$ 0.300 Btu-in/hr-ft^2-°F is attainable with a batt comprised entirely of 12 micron diameter (approximately 1.5 denier) polyester fiber (for current, practical purposes, the polyester data shown is also applicable to equal-density batts made from other fibrous materials). The 0.300 Btu-in/hr-ft^2-°F and 12 micron co-ordinates thus established an upper limit for primary fiber diameter in the FR batts to be developed. They also indicated that the primary fiber diameter should be somewhat less than 12 microns to compensate for inclusion of larger diameter fiber, particularly binder fiber, in the blend. This rationale was further supported by previously cited experience.

Table 1. ***Fiber Candidates and Anticipated Attributes for Which Each was Selected***

Fiber Candidate, Manufacturer	Cost[a] ($/lb)	Cost[a] ($/kg)	Linear Density (denier)	Linear Density (dtex)	Effective[b] Diameter (microns)	Anticipated Attributes
Nomex aramid, DuPont	10.30	22.69	1.5	1.7	12	Good FR; marginal diameter; acceptable cost
Kevlar aramid, DuPont	11.50	25.33	1.5	1.7	12	Similar to Nomex (above), but slightly greater cost; potential availability advantage over Nomex
Kynol novoloid, American Kynol	8.00	17.62	2.0	2.2	15	Good FR; diameter greater than preferred; relatively low cost
P-84 polyimide with hydrophobic finish, Lenzing	24.00	52.86	0.55	0.61	8	Good FR; near optimal diameter; producer applied WR; relatively high cost
P-84 polyimide with hydrophobic finish, Lenzing	15.00	33.04	1.5	1.7	13	Good FR; marginal diameter; producer applied WR; cost advantage over 0.55 denier (0.61 dtex) P-84
FR viscose, Lenzing	4.75	10.46	1.5	1.7	12	FR uncertain; marginal diameter; low cost
Ryton polyphenylene sulfide, Amoco (ex-Phillips)	8.40	18.50	1.8	2.0	14	Good FR; diameter greater than preferred; relatively low cost
Polybenzimidazole (PBI), Hoechst Celanese	61.50	135.46	1.5	1.7	12	Good FR; marginal diameter; high cost
FR polyester, standard finish, Hoechst Celanese	1.50	3.30	1.5	1.7	12	Acceptable FR; marginal diameter; major cost advantage
FR polyester with hydrophobic finish, Hoechst Celanese	1.75	3.85	1.5	1.7	12	Acceptable FR; marginal diameter; producer applied WR; major cost advantage
Bicomponent binder fibers, several types, Hoechst Celanese	1.50	3.30	4.0	4.4	20	This class of binder fiber proven in similar, but non-FR, applications.

a. Cost at the outset of the program, when fibers were selected.
b. Not all fiber candidates have round cross-sections; effective diameter calculated from denier and specific gravity, assuming round cross-section.

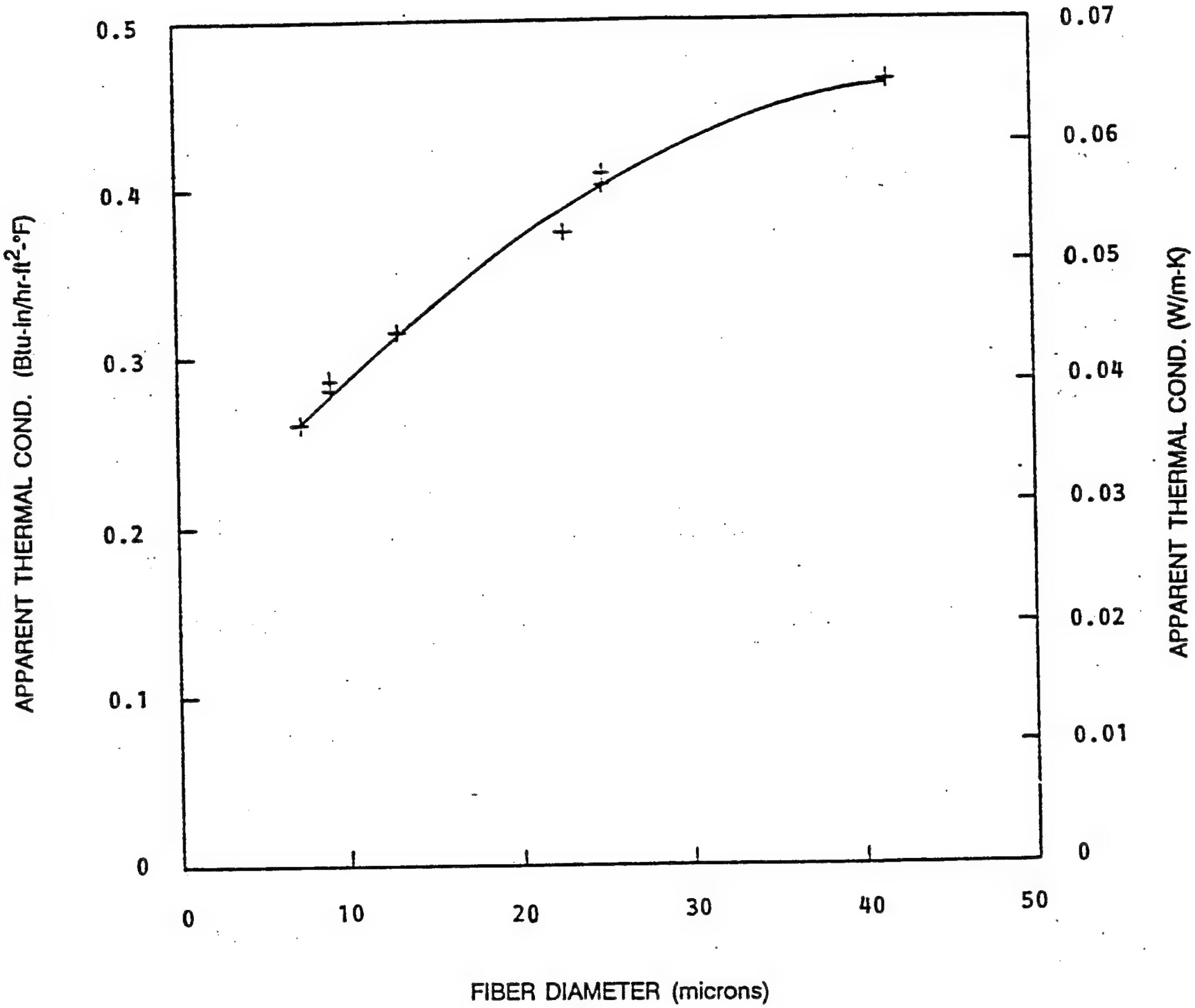

Figure 1. *Apparent Thermal Conductivity as a Function of Fiber Diameter for Polyester Insulators. All had a density of approximately 0.5 lb/ft^3 and were about 2 inches thick; measurements made by plate-to-plate method, ASTM C518; heat flow was downward*

C. Flammability Test Screening

Application of the various resources available for fiber selection (discussed above) provided confidence that most of the insulator properties sought were attainable through use of any of several fiber candidates. However, a singularly important exception was concern that flame resistance requirements might not easily be met in the very low density (0.3 to 0.6 lb/ft^3) batt configuration desired. Flammability reference data was available for most of the fiber candidates, although test conditions were infrequently equivalent and data for low density batts was virtually non-existent. Consequently, flammability test screening of low density batts made with each fiber candidate was an essential first laboratory task. Vertical flammability testing per Method 5903 of Federal Standard 191A and horizontal and inclined testing per Method 5907 were both specified in the contract Work Statement, but preliminary work showed that passing the vertical test of Method 5903 was substantially more difficult for typical samples of interest. The vertical flammability test thus became the preferred screening tool.

Initial screening for vertical flammability characteristics, as well as for several other performance properties, was work that would provide data for both staple and continuous filament selection. All of the trial batts, however, were fabricated using staple fiber. Staple can be opened, blended and made into batts, on a laboratory scale, whereas the nature of continuous filament tow processing makes small-scale, frequent trials virtually impossible.

Laundering durability and minimum density objectives for the insulators to be developed made use of relatively new, thermoplastic, bicomponent, binder fiber technology a sound approach. Bicomponent binder fiber had provided successful solutions, especially in terms of conflicting durability and low density requirements, in recent, similar insulator applications[3,4], although they were ones in which flame resistance was not an issue. This effort began with the intent of employing bicomponent binder fiber, in both staple and continuous filament insulators, if the anticipated negative effect of thermoplastic binder on flammability was, or could be made, acceptable. Binder fiber was expected to interact with various FR fiber candidates in different ways during flame exposure and so it was included in most batt samples made for vertical flammability test screening. The binder fiber used initially was Hoechst Celanese Type K54 (polyester/polyester, high melt/low melt, sheath/core), in the proportion of 82% FR candidate/18% binder, by weight. Recent work had shown that this binder and weight fraction constituted a plausible, preliminary combination.

The first vertical flammability tests for screening FR fiber candidates were made with bonded batts that had been fabricated on the bench top. A laboratory opener/blender, Model 338, manufactured by

Spinlab, Inc. of Knoxville, Tennessee facilitated the make up of uniform batt samples. Recognition of the potential effect of batt thickness and density upon flammability, together with awareness of the dimensional and weight requirements of the insulators to be developed, led to adoption of these standard specifications for test batts: (1) thickness of 0.67 inches, (2) areal density of 4 oz/yd^2, and (3) volume density of 0.50 lb/ft^3. Shrinkage during thermal bonding of the samples made absolute control over all three interactive parameters difficult, but technique adjustments for each fiber type eventually yielded samples that were acceptably close in terms of weight and dimensions.

The results of vertical flammability screening tests for FR fiber candidates are reported in Tables 2 and 3. Table 2 consists of data for eight inherently FR fiber types and Table 3 contains data for several polyester variants, including some with a phosphorus based additive to provide flame resistance (designated FR in table). None of the eight fiber types of Table 2 were completely excluded from further consideration on the basis of the data shown. Fiber finish or other surface contaminants were suspected to be responsible for one or two unusually long afterflame and afterglow times within otherwise acceptable data sets and, although not all fiber candidates met all vertical flammability targets, few data points were far beyond target values. However, the FR polyester data of Table 3 contained many data points that did lie well beyond target values. The Table 3 data showed that:

1. The phosphorus based FR additive did not significantly reduce char length and char lengths were generally on the order of twice the 3.5 inch target value.

2. The water repellent finish employed, a polydimethylsiloxane, had a negative effect, especially upon afterflame times.

3. Increasing batt density to about 1 lb/ft^3 did not significantly reduce afterflame times or char length.

Program plans, made during the proposal stage, relied upon using FR polyester, either as a primary or as a secondary fiber component, to reduce insulator cost without significantly compromising performance. This polyester role was endorsed by the program sub-contractor, Hoechst Celanese, who was to provide FR polyester for both staple and continuous filament insulator configurations. The Table 3 data, however, cast some doubt upon FR polyester's utility.

Table 2. ***Results of Vertical Flammability Screening Tests[a] Made on a Series of Two-Component, Spinlab Batts***

Primary Fiber Component[b]	Linear Density, Primary Fiber		Flammability Test Results					
	Denier	Dtex	Test Direction	After-flame (sec)	After-glow (sec)	Char Length (inch)	Char Length (cm)	Pass/Fail
Program Target	--	--	--	0	≤25	≤3.5	≤8.9	
PBI, 82%	1.5	1.7	MD MD XD	0 0 0	6 23 2	0 0.5 0	0 1.3 0	Pass
Kevlar, 82%	1.5	1.7	MD MD MD MD MD XD XD XD	0 43 0 0 0 0 0 0	1 0 2 2 3 1 3 2	0 0.8 0.1 0.1 0.2 0 0.1 0.2	0 2.0 0.2 0.2 0.5 0 0.2 0.5	Tentative pass[c]
Nomex, 82%	1.5	1.7	MD MD XD	3 13 0	3 0 0	2.5 4.8 3.0	6.4 12.2 7.6	Tentative pass[c]
Kynol, 82%	2.0	2.2	MD MD MD MD XD XD	0 0 0 0 0 0	3 2 50 45 6 2	0.5 0.9 0.5 0.6 0.4 0.7	1.3 2.3 1.3 1.5 1.0 1.8	Tentative pass[c]
FR Viscose, 82%	1.5	1.7	MD MD XD	0 0 0	2 0 2	4.4 6.0 3.0	11.2 15.2 7.6	Fail
Ryton, 82%	1.8	2.0	MD MD XD	0 0 0	0 0 0	6.2 5.7 4.3	15.7 14.5 10.9	Fail
P84 microfiber, WR finish, 82%	0.55	0.61	MD XD	3 6	3 6	3.0 2.4	7.6 6.1	Tentative pass[c]
P84, WR finish, 82%	1.5	1.7	MD XD	0 0	2 2	3.1 3.3	7.9 8.4	Pass

a. Per Method 5903.1, "Flame Resistance of Cloth, Vertical," of Fed. Std. No. 191A. All samples were 4 oz/yd^2 (135 g/m^2) bonded batts of 0.67 inch (1.70 cm) thickness (nominal; variation existed), with a nominal volume density of 0.50 lb/ft^3 (8.0 kg/m^3).

b. In every case, the secondary fiber component was Hoechst Celanese binder fiber, Type K54.

c. Finishes or other surface contaminants suspected of influencing results.

Table 3. ***Results of Vertical Flammability Screening Tests[a] Made on a Series of Two-Component and Single Component Polyester Batts Prepared Using a Spinlab Blender***

Primary Fiber Component[b]	Average Volume Density (lb/ft^3)	Average Volume Density (kg/m^3)	Flammability Test Results: Test Direction	After-flame (sec)	After-glow (sec)	Char Length (inch)	Char Length (cm)	Pass/Fail
Program Target	--	--	--	0	≤25	≤3.5	≤8.9	--
Polyester, without FR and without WR finish, 82%	0.50	8.0	MD	0	0	5.7	14.5	
			MD	0	0	5.0	12.7	
			XD	0	0	6.1	15.5	Fail
Polyester, without FR and with WR finish, 82%	0.50	8.0	MD	13	0	8.0	20.3	
			MD	20	0	6.0	15.2	
			XD	23	0	7.0	17.8	Fail
Polyester, with FR and without WR finish, 82%	0.50	8.0	MD	0	0	6.0	15.2	
			MD	0	0	7.0	17.8	
			MD	0	0	4.8	12.2	
			MD	0	0	8.0	20.3	
			MD	0	0	6.4	16.2	
			MD	0	0	9.0	22.9	
			XD	0	0	5.1	13.0	
			XD	0	0	4.8	12.2	
			XD	0	0	5.0	12.7	Fail
	1.06	17.0	MD	0	0	5.5	14.0	
			MD	0	0	5.3	13.5	
			XD	0	0	5.8	14.7	Fail
Polyester, with FR and with WR finish, 82%	0.50	8.0	MD	0	2	7.0	17.8	
			MD	0	0	4.6	11.7	
			MD	0	0	4.0	10.2	
			MD	16	0	6.5	16.5	
			MD	2	0	9.0	22.9	
			MD	13	0	7.0	17.8	
			XD	0	6	5.0	12.7	
			XD	0	0	5.5	14.0	
			XD	3	0	6.0	15.2	
			XD	15	0	7.0	17.8	
			XD	4	0	9.0	22.9	Fail
	0.84	13.5	MD	22	0	6.8	17.3	
			MD	17	0	5.0	12.7	
			XD	10	0	6.0	15.2	Fail
	0.87	13.9	MD	18	0	6.0	15.2	
			MD	18	0	6.3	16.0	
			XD	25	0	7.0	17.8	Fail
	1.14	18.3	MD	18	0	5.5	14.0	
			MD	29	0	5.8	14.7	
			XD	0	0	7.0	17.8	Fail
FR polyester, 100% (no binder)	0.50	8.0	MD	0	0	7.5	19.0	Fail
FR polyester, WR finish, 100% (no binder)	0.50	8.0	MD	2	0	8.0	20.3	Fail
Binder fiber only (100%) Hoechst Celanese Type 54	0.50	8.0	MD	0	0	5.7	14.5	
			XD	0	0	5.5	14.0	Fail

a. Per Method 5903.1, "Flame Resistance of Cloth, Vertical," of Fed. Std. No. 191A. All samples were 4 oz/yd^2 (135 g/m^2) bonded batts (nominal; variation existed). Thicknesses varied as a function of volume density.

b. The secondary component in all two-component batts was Hoechst Celanese binder fiber, Type K54.

D. Issues Specific to Continuous Filament Selection

At this juncture, several viable fiber candidates appeared to be available for the staple insulator approach. However, during the fiber sample procurement process it had become apparent that only two fiber producers would be able to deliver continuous filament tow. One of these was Phillips Fibers (now Amoco), producers of Ryton (polyphenylene sulfide, a thermoplastic, and one of the less desirable fibers on the basis of the vertical flammability data of Table 2); the other was Hoechst Celanese, who had previously agreed to provide FR polyester tow and tow spreading expertise as a sub-contractor. The Ryton tow made for the program by Phillips was not suitable for opening and spreading. The tow ribbon was not adequately crimped, lacked cohesion and was characterized by many tangles that prevented further processing. Although Phillips expressed continuing interest in developing a Ryton tow for opening and spreading, it was apparent, early in the program, that this could not be counted upon. In fact, the tow described above later proved to be the only one they would provide.

The relatively poor vertical flammability performance of FR polyester test batts and the lack of a continuous filament alternative to FR polyester became the focus of several conferences between us (Albany International Research) and our sub-contractor (Hoechst Celanese). During these conferences, lack of agreement on char length interpretation became an impediment to progress. We interpreted char length rather strictly, measuring all shrunken-away void area on the sample, whether blackened or not, as char (all data reported herein is based upon this kind of assessment). Hoechst Celanese maintained that this did not yield a fair representation of the performance of their FR polyester, which does not, in fact, burn and char, but melts and shrinks away from the flame. The importance of this issue led to a meeting at Natick on September 29, 1992. It was attended by Ms. Margaret Auerbach, Ms. Rita Devarakonda and Mr. Richard Lacerte of Natick; Messrs. Robert Averell, Kevin Campbell and Eugene Steadman and Ms. Elizabeth van Amerongen of Hoechst Celanese; and Dr. Maryann Kenney, Ms. Cynthia Egan and Mr. James Donovan of Albany International Research. The results of this meeting were as follows:

1. Natick agreed with Albany International Research's interpretation of the char length, i.e., the char length should include all shrunken-away void areas on the sample.

2. All Hoechst Celanese, Albany International and Natick participants agreed that FR polyester was the only viable candidate for making continuous filament, flame-resistant, insulating batts.

3. Most participants agreed that better choices than FR polyester were available for the primary component in staple-based, flame-resistant, insulating batts.

4. Ms. Auerbach pointed out that the Army had no flame-resistant, continuous filament, insulating material available for sleeping bag applications and so adapting FR polyester for this purpose would constitute distinct improvement. She also related that the Army employed inherently FR fiber such as aramid and Kynol in certain staple-based clothing insulators and was seeking to improve overall performance in these applications. Thus, Ms. Auerbach pointed out, the Army's FR, continuous filament insulator needs differed from their FR, staple insulator needs. She then suggested that modification of the contract's char length objectives, for the continuous filament insulator only, would facilitate the best possible outcome for both continuous filament and staple FR insulator development efforts.

Subsequently, the Government formally modified the flame resistance objectives for the continuous filament insulator. These modified objectives are shown below in Table 4, together with the original, unchanged objectives for the staple insulator.

Table 4. ***Flammability Target Values, as Modified (Contract Modification P00001), to be Measured by Methods 5903 and 5907 of Federal Standard 191A, Before and After Laundering***

Insulator Type	Afterflame (sec)	Afterglow (sec)	Char Length[a]		Additional Requirement
			(inch)	(cm)	
Staple	0	≤25	≤3.5	≤8.9	No flame propagation, melting or dripping
Continuous Filament	≤2	≤25	≤5.5[a]	≤14.0	No flaming melt drip

a. "No individual specimen measuring more than 6.5 inch (16.5 cm). Ten specimens from both the machine and cross-machine direction shall be tested - the two directions shall not be averaged together."

E. Conclusion

The Natick meeting and subsequent contract modification served to finalize the choice of FR polyester for use in the continuous filament insulator, although events up to that point had contributed a sense of inevitability to the matter.

Initial flammability screening of fiber candidates provided assurance that a significantly improved staple insulator could be developed, but it did not result in elimination of staple fiber candidates. Finishes and surface contaminants had apparent adverse effects upon the performance of several fiber types and each required further attention before the staple candidate list could be ranked or reduced in length. Work toward this end and the selection of the preferred staple fiber blend is the subject of the following section.

3. STAPLE INSULATOR DEVELOPMENT

A. Introduction

The staple insulator development process followed the experimental approach described previously, i.e., existing knowledge was utilized to the greatest extent possible and experimentation was sequentially directed toward addressing the next most difficult insulator performance objective, in turn. Fiber blending offered opportunity to utilize desired attributes of more than one fiber type in striving to meet performance targets. Responding to the opportunity, however, required many blending/batt-making/testing steps, since the predictability of some blended batt properties, especially flame resistance, is poor. Staple insulator development thus consisted of a reiterative experimental series which was continually redirected on the basis of new data analysis. As work progressed, the fiber candidate list grew shorter and less difficult insulator objectives received attention. Eventually, only the most viable fiber candidates remained and all performance objectives had been addressed.

B. Flammability Test Screening of Blends

Three-component (including binder) blends appeared to offer the best compromise between utility and manageability and so work began, and ultimately remained focused upon, three-component blends. In most cases, the three-component blends included: (1) a high performance, relatively costly fiber, (2) an adequately performing fiber included primarily to reduce the cost of the blend and (3) a binder fiber.

Initial vertical flammability tests made to screen fiber candidates (reported in the previous section) reinforced the need to concentrate first on vertical flammability requirements. The initial tests also provided data that was used to select the first three-component blends for evaluation. These blends generally consisted of the following:

1. An FR fiber that had yielded promising results in initial flammability screening, as reported in Table 2, Section II.

2. Hoechst Celanese FR polyester, 1.5 denier, with WR finish <u>or</u> Fiber Industries polyester, 0.5 denier (without FR), with WR finish, and

3. Hoechst Celanese Type K54 polyester binder fiber, 4 denier.

The three-component blends were made into 0.5 lb/ft^3 test batt samples and tested in accord with Method 5903 of Federal Standard 191A. The results are given in Table 5. Several of the three-component sample types that generally performed well in this test series also exhibited behavior that suggested caution. This behavior was of two types: (1) infrequent afterflame and (2) a tendency to "flash" or "flash-over" sporadically. This latter, "flashing," phenomenon not only occurred sporadically, but varied greatly when it did occur. It appeared to be the rapid burning of exposed fiber tips on the lofty, loose-surfaced batts and affected only the most outer surfaces, leaving a trace of surface char, often on samples that otherwise easily met the program's vertical flammability requirements.

Acknowledgement of the potential importance of the sporadic afterflame and flash phenomena led to several experiments directed toward obtaining an understanding of factors that influenced them. These experiments relied upon comparing vertical flammability results for batts that differed only in one fiber component, the difference in that component usually being the fiber surface, as changed by scouring or by addition of a water repellent. The result of these experiments are reported in Tables 6 and 7.

The data of Table 6, which shows the effect of fiber scouring upon the vertical flammability resistance of batts made of P84 microfiber and of Nomex, shows that scouring eliminated afterflame in both cases. This finding was not wholly conclusive due to the minimal number of tests performed and because of the random nature of afterflame. It was, nonetheless, useful as subsequent experimental steps were planned. Flash was not observed in any of the tests reported in Table 6.

The Kevlar and Kevlar / polyester batts for which vertical flammability data is reported in Table 7 were the first prepared using a 12-inch machine card. All data reported in preceding tables was for smaller batt samples prepared using a Spinlab benchtop opener/blender. The results of Table 7 suggested that the Kevlar (41%) / FR polyester (41%) / binder (18%) blend, which was the primary subject of the table, may have been acceptable. All variants, regardless of fiber surface condition, did not exhibit afterflame and easily met afterglow and char length objectives. However, the issue of flash, which was a consideration in planning the experiments that provided the Table 7 data, was not still adequately understood. It occurred infrequently during the testing reported in Table 7, with one exception. The exception, sample batts of 100% scoured Kevlar, which flashed to some degree in every one of ten individual tests, provided helpful evidence. The 100% scoured Kevlar batts were unique among the Table 7 sample set for several reasons: (1) they did not include a polyester component, (2) their fiber surfaces were presumably free of contamination, and (3) because they were not bonded and of relatively low density, their surfaces were very "hairy." (The scoured, 100% Kevlar batt samples were, due to an additional opening step, more "hairy" than the 100%, as-received, Kevlar samples.) These characteristics of the 100% scoured Kevlar samples strongly indicated that the nature of the batt surface was important to the flash phenomenon.

Table 5. *Results of Vertical Flammability Tests[a] Made on a Series of Three-Component Batts Prepared Using a Spinlab Blender*

Fiber Components			Flammability Test Results					
A	B[b]	C[c]	Test Direction	After-flame (sec)	After-glow (sec)	Char Length (inch)	Char Length (cm)	Pass/Fail, Other Comments
Program Target			--	0	≤25	≤3.5	≤8.9	
Kevlar (41%)	FR polyester, WR finish (41%)	Binder (18%)	MD MD XD	0 0 0	5 0 3	0.1 0.2 0	0.2 0.5 0	Pass; Also see Table 7
Kevlar (20%)	FR polyester, WR finish (62%)	Binder (18%)	MD MD MD XD XD	0 0 0 0 7	2 0 20 2 0	1.9 0.2 0.2 2.0 0.2	4.8 0.5 0.5 5.1 0.5	Flash-over, 3 samples; tentative pass
Kevlar (41%)	0.5 den (0.55 dtex) polyester, WR finish (41%)	Binder (18%)	MD MD MD MD XD XD	0 6 16 0 0 8	3 0 0 3 3 0	0.2 0.5 0.5 0.2 0.4 0.2	0.5 1.3 1.3 0.5 1.0 0.5	Flash-over, all samples; fail
Kevlar (20%)	0.5 den (0.55 dtex) polyester, WR finish (62%)	Binder (18%)	MD MD XD	4 17 6	0 0 0	0.5 0.5 1.5	1.3 1.3 3.8	Flash-over, all, ignited tops; fail
Kynol (41%)	FR polyester, WR finish (41%)	Binder (18%)	MD MD MD MD XD XD	0 0 0 0 0 0	3 4 2 2 4 3	0.4 0.5 0.3 0.2 0.5 0.7	1.0 1.3 0.8 0.5 1.3 1.8	Pass
Kynol (20%)	FR polyester, WR finish (62%)	Binder (18%)	MD XD	0 13	2 0	5.2 4.5	13.2 11.4	Fail
Kynol (41%)	0.5 den (0.55 dtex) polyester, WR finish (41%)	Binder (18%)	MD XD	0 0	3 3	2.7 3.3	6.9 8.4	Pass
P84, 0.55 den (0.61 dtex), WR finish (41%)	FR polyester, WR finish (41%)	Binder (18%)	MD MD XD	0 12 0	0 0 0	9.0 6.8 9.0	22.9 17.3 22.9	Fail
PBI (41%)	FR polyester, WR finish (41%)	Binder (18%)	MD MD XD XD	5 6 0 3	0 0 0 0	0.5 0.5 0.5 2.0	1.3 1.3 1.3 5.1	Fail
PBI (20%)	FR polyester, WR finish (62%)	Binder (18%)	MD MD XD	4 4 4	0 0 0	2.5 1.0 3.0	6.4 2.5 7.6	Fail

a. Per Method 5903.1, "Flame Resistance of Cloth, Vertical," of Fed. Std. No. 191A. All samples were 4 oz/yd^2 (135 g/m^2) bonded batts of 0.67 inch (1.70 cm) thickness (nominal; variation existed), with a nominal volume density of 0.50 lb/ft^3 (8.0 kg/m^3).

b. All FR polyester cited in this column, with and without WR (Si water repellent), was 1.5 denier (1.7 dtex) product supplied by Hoechst Celanese.

c. The binder fiber used in all samples was: Hoechst Celanese Type K54, 4 denier (4.4 dtex).

Table 6. ***Results of Vertical Flammability Tests[a] Made to Evaluate Differences Due to Fiber Finish and/or Surface Contaminants***

Primary Fiber Component[b]	Flammability Test Results					
	Test Direction	After-flame (sec)	After-glow (sec)	Char Length (inch)	Char Length (cm)	Pass/Fail
Program Target	--	0	≤25	≤3.5	≤8.9	
P84 microfiber, WR finish, 82%[c]	MD XD	3 6	3 6	3.0 2.4	7.6 6.1	Fail
Scoured[d] P84 microfiber, 82%	MD XD	0 0	8 4	1.9 3.4	4.8 8.6	Pass
Nomex, 82%[c]	MD MD XD	3 13 0	3 0 0	2.5 4.8 3.0	6.4 12.2 7.6	Fail
Scoured[d] Nomex, 82%	MD XD	0 0	22 13	2.0 1.6	5.1 4.1	Pass

a. Test method and sample configuration as given in Table 5, footnote a.
b. The secondary component in all two-component batts was Hoechst Celanese binder fiber, Type K54, 4 denier (4.4 dtex).
c. From Table 2.
d. Scoured with a 0.1% basic solution of sodium hydroxide, then neutralized in a two-step process using 0.25% sodium carbonate solution first, followed by a rinse in 0.5% acetic acid solution.

Table 7. ***Results of Vertical Flammability Tests[a] Made on Kevlar and Kevlar/Polyester Batts to Evaluate Differences Due to Fiber Surface Condition***

Fiber Components			Average Volume Density		Flammability Test Results				
					Test Direction	After-flame (sec)	After-glow (sec)	Char Length	
A	B	C[c]	(lb/ft^3)	(kg/m^3)				(inch)	(cm)
Program Target						0	≤25	≤3.5	≤8.9
Kevlar, as received (41%)	FR polyester, WR finish (41%)	Binder (18%)	0.50	8.0	MD	0	2	0.2	0.5
					MD	0	2	0.2	0.5
					MD[d]	0	2	0.2	0.5
					XD	0	2	0.2	0.5
					XD	0	2	0.1	0.2
					XD	0	2	0.2	0.5
					XD	0	2	0.2	0.5
Kevlar, Scoured[b] (41%)	FR polyester, WR finish (41%)	Binder (18%)	0.50	8.0	MD[f]	0	2	0.2	0.5
					MD	0	2	0.2	0.5
					MD	0	2	0.2	0.5
					XD	0	2	0.1	0.2
					XD	0	2	0.2	0.5
					XD	0	2	0.1	0.2
					XD	0	3	0.2	0.5
Kevlar, as received (41%)	FR polyester, without WR finish (41%)	Binder (18%)	0.50	8.0	MD[f]	0	3	0.2	0.5
					MD	0	2	0.2	0.5
					MD	0	2	0.2	0.5
					XD[d]	0	2	0.2	0.5
					XD[d]	0	2	0.1	0.2
					XD	0	2	0.2	0.5
					XD	0	3	0.2	0.5
Kevlar, Scoured[b] (41%)	FR polyester, without WR finish (41%)	Binder (18%)	0.50	8.0	MD	0	2	0.2	0.5
					MD	0	3	0.2	0.5
					MD	0	3	0.2	0.5
					XD	0	3	0.2	0.5
					XD	0	2	0.2	0.5
					XD	0	2	0.2	0.5
					XD	0	2	0.2	0.5
Kevlar, as received (100%)			0.27	4.3	MD	0	2	0.5	1.3
					MD	0	3	0	0
					MD	0	3	0.1	0.2
					XD	0	3	0.1	0.2
					XD	0	2	0.1	0.2
					XD[h]	0	3	0.2	0.5
Kevlar, Scoured[b] (100%)			0.34	5.4	MD[d]	0	3	0.2	0.5
					MD[d]	0	4	0.2	0.5
					MD[d]	0	3	0.1	0.2
					MD[e]	0	3	0.2	0.5
					MD[d]	0	2	0.1	0.2
					XD[f]	0	3	0.1	0.2
					XD[g]	0	3	0.1	0.2
					XD[g]	0	3	0.1	0.2
					XD[g]	0	2	0.1	0.2
					XD[g]	0	3	0.1	0.2

a. Per method 5903.1, "Flame Resistance of Cloth, Vertical," of Fed. Std. No. 191A.
b. Scour solution of 1% Merpol HCS heated to 160°F to 180°F (71° to 82°C) for 30 minutes; multiple water rinses.
c. The binder fiber used in all samples was Hoechst Celanese Type K54, 4 denier (4.4 dtex).
d. Flash to top of sample before 12 second test started.
e. Flash on both sides of sample before test started.
f. Flash to top of sample before extinguishing.
g. Minor flash.
h. Flash half way up sample sides before extinguishing.

C. Expanded Testing with Five Primary Fiber Candidates

At this point in the staple insulator development effort, the number of fiber candidates had been reduced from ten to five, based primarily upon vertical flammability test results for two-component and three-component (both including binder) blended batts (the first of these results was reported in previous Section II). However, unresolved issues left the ultimate role of each of the remaining five candidates undetermined, as summarized below:

1. FR polyester with a water repellent finish remained attractive as a cost-reducing component, and exhibited acceptable FR performance in some blends. However, its effect on FR performance remained suspect.

2. Kevlar generally yielded acceptable FR performance except that some degree of surface flash was observed during most vertical flammability test series of Kevlar-containing batts. Kevlar was not available with producer-applied water repellent. It should be noted that Kevlar had been selected for further evaluation, and Nomex eliminated, because Kevlar/binder blends exhibited char lengths on the order of only 0.2 inch, compared to char lengths of 2.5 to 4.8 inches for comparable Nomex blends (the program target was ≤3.5 inch). Additionally, afterflame was generally not a problem with Kevlar blends, but afterflame times of up to 13 seconds were measured for Nomex blends (the program target was 0 second). Scouring Nomex, which is not a practical production option, eliminated afterflame, but increased afterglow times to 13 and 22 seconds (two samples only; the program target was ≤25 seconds).

3. Kynol, in two different polyester blends, performed acceptably in limited (by sample quantity) vertical flammability testing. However, Kynol's linear density of 2.0 denier indicated that it had the largest cross-sectional area of all fiber candidates and would require blending with fiber of very small cross-sectional dimension to produce a batt with acceptable insulating efficiency. Kynol was not available with producer-applied water repellent.

4. P84 microfiber (0.55 denier) with producer-applied water repellent finish had been blended with binder (82/18) to make a two-component batt that unexpectedly failed to meet the program after-flame target. Subsequently, a P84 microfiber / FR polyester blend also exhibited similarly poor FR test results. Correspondence with the fiber manufacturer, Lenzing AG, led to discovery of excess finish application to which Lenzing attributed the poor FR performance. Our FR evaluation of scoured fiber samples supported their conclusion and indicated that the fiber could provide the FR performance desired.

5. P84 macrofiber (1.5 denier) with producer-applied water repellent finish performed acceptably in limited FR evaluations. However, the finish application problem discovered in work with P84 microfiber proved to be common to the macrofiber and so experimental work with it had been intentionally limited up to this point.

In terms of the specific requirements of the program, the FR performance of the five remaining fiber candidates was now understood reasonably well, but without the complete confidence required to reduce the list further. However, the plausibility of each of the five candidates began to make assessment or confirmation of other performance characteristics important; minor differences in FR performance might soon need to be balanced against other parameters. Consequently, another round of sample-batt making, to provide specimens for an expanded test series, began. Using a 12-inch wide laboratory card, blended, bonded batt samples of the following compositions were made:

1. Kevlar, 1.5 denier (41%) / Hoechst Celanese polyester, 1.5 denier, FR, WR (41%) / K54 binder fiber (18%).

2. Kynol, 2.0 denier (41%) / Hoechst Celanese polyester, 1.5 denier, FR, WR (41%) / K54 binder fiber (18%).

3. P84, 1.5 denier, WR (41%) / Hoechst Celanese polyester, 1.5 denier, FR, WR (41%) / K54 binder fiber (18%).

4. P84, 0.55 denier, WR (82%) / K54 binder fiber (18%).

The first three of these samples were regarded as feasible three-component candidates and the fourth was included for comparison with data that was available for a like blend with optimal WR finish. The P84 macrofiber (1.5 denier) of blend 3., above, was suspected of having excess WR finish; the P84 microfiber (0.55 denier) of blend 4. was believed to be as desired in terms of finish level. Three types of tests were performed on the batt samples to determine:

1. Vertical flammability characteristics, per Method 5903 of Federal Standard 191A

2. Flammability characteristics per the methenamine pill test, Method 5907 of Federal Standard 191A, two variations

3. Thermal conductivity per ASTM C518, using the Rapid-K apparatus

4. Water repellency in terms of water absorption after 20 minutes of immersion

The water repellency measurements with the trial batts were expanded to include measurements with a variety of opened, loose fiber samples. The loose fiber sample set included fiber in as-received condition, scoured fiber, and fiber to which we applied water repellent finish.

Data from the test series outlined above, reported in Tables 8 through 13, lead to observations and conclusions that were eventually applied to final fiber and blend selection. The most significant of these observations and conclusions were:

1. The surface flash phenomenon previously observed in many Kevlar blend tests occurred in all ten vertical flammability tests of the Kevlar / FR polyester / binder (41/41/18) blended batt (Table 8). Neither the cause of nor the full significance of the flash phenomenon to end-items could be determined within the scope of the program, necessitating a cautious approach toward this clearly negative characteristic. At this point, we decided to eliminate Kevlar from consideration as a primary FR component.

2. The vertical flammability performance of blended, bonded batts consisting of Kynol / FR polyester/ binder (41/41/18) and of P84 macrofiber / FR polyester / binder (41/41/18) was disappointingly poor (Table 8), providing strong evidence that the ultimate blend should not contain a major fraction of FR polyester.

3. The methenamine-pill test data of Tables 9 and 10 generally supported the above conclusions based upon vertical flammability testing and certainly did not provide contradictory evidence.

4. The thermal conductivity data of Table 11 showed that blends containing primarily 1.5 denier fiber, with some 4 denier binder fiber (18%), have thermal conductivities approximately equal to the program's upper target limit of 0.300 Btu-in/hr-ft^2-°F. It was then decided to reduce the thermal conductivity of the final candidate blend to a value somewhat below 0.300 Btu-in/hr-ft^2-°F by including a smaller-diameter fiber component.

Table 8. *Results of Vertical Flammability Tests[a] Made on a Series of Multi-component Batts Prepared Using a 12-inch (30-cm) Machine Card*

Fiber Components			Flammability Test Results					
						Char Length		
A	B[b]	C[c]	Test Direction	Afterflame (sec)	Afterglow (sec)	(inch)	(cm)	Pass/Fail, Other Comments
Program Target			–	0	≤25	≤3.5	≤8.9	
Kevlar (41%)	FR polyester, WR finish (41%)	Binder (18%)	MD	0	3	0.2	0.5	
			MD	0	4	0.2	0.5	
			MD	0	4	0.2	0.5	
			MD	0	4	0.2	0.5	
			MD	0	4	0.2	0.5	
			MD, Avg.	0	4	0.2	0.5	
			XD	0	3	0.1	0.2	
			XD	0	3	0.1	0.2	
			XD	0	4	0.1	0.2	
			XD	0	4	0.1	0.2	
			XD	0	4	0.2	0.5	Fail; every sample
			XD, Avg.	0	4	0.1	0.2	flashed over.
Kynol (41%)	FR polyester, WR finish (41%)	Binder (18%)	MD	52	2	9.0	23	
			MD	40	2	9.0	23	
			MD	4	3	3.8	9.6	
			MD	27	3	9.0	23	
			MD	28	2	9.0	23	
			MD, Avg.	30	2	8.0	20	
			XD	21	2	9.0	23	
			XD	25	2	9.0	23	
			XD	22	2	9.0	23	
			XD	18	2	9.0	23	
			XD	19	2	9.0	23	
			XD, Avg.	21	2	9.0	23	Fail
P84, 1.5 den (1.7 dtex), WR finish (41%)	FR polyester, WR finish (41%)	Binder (18%)	MD	27	1	9.0	23	
			MD	26	0	9.0	23	
			MD	25	0	9.0	23	
			MD	26	0	9.0	23	
			MD	20	0	9.0	23	
			MD, Avg.	25	0	9.0	23	
			XD	26	0	9.0	23	
			XD	28	0	9.0	23	
			XD	32	1	9.0	23	
			XD	23	1	9.0	23	
			XD	21	1	9.0	23	
			XD, Avg.	26	1	9.0	23	Fail
P84, 0.55 den (0.61 dtex), WR finish (82%)	None	Binder (18%)	MD	0	2	1.5	3.8	
			MD	0	2	1.4	3.6	
			MD	0	4	1.0	2.5	
			MD	0	3	1.2	3.0	
			MD	0	8	1.7	4.3	
			MD, Avg.	0	4	1.4	3.6	
			XD	0	6	1.3	3.3	
			XD	0	3	1.6	4.1	
			XD	0	3	1.3	3.3	
			XD	0	2	1.1	2.8	
			XD	0	2	1.6	4.1	
			XD, Avg.	0	3	1.4	3.6	Pass
Pyroloft® manufactured by Albany/Lenzing Venture; essentially the same as the P84 0.55 den (0.61 dtex) blend immediately above.			MD	0	2	0.7	1.8	
			MD	0	2	1.0	2.5	
			MD	0	1	1.0	2.5	
			MD	0	2	1.5	3.8	
			MD	0	3	1.1	2.8	
			MD, Avg.	0	2	1.1	2.8	
			XD	0	4	1.0	2.5	
			XD	0	3	1.2	3.0	
			XD	0	2	1.5	3.8	
			XD	0	1	1.0	2.5	
			XD	0	2	1.0	2.5	
			XD, Avg.	0	3	1.2	3.0	Pass

a. Test Method and sample configuration as given in Table 5, footnote a.
b. All FR polyester cited in this column, with WR (Si water repellent), was 1.5 denier (1.7 dtex) product supplied by Hoechst Celanese.
c. The binder fiber used in all samples was: Hoechst Celanese Type K54, 4 denier (4.4 dtex).

Table 9. ***Results of Folded Batt, Methenamine Pill, Flammability Tests (Fed. Std. 191A, Method 5907) Made on a Series of Multicomponent Batts Prepared Using a 12-inch (30-cm) Machine Card***

Fiber Components[a]			Flammability Test Results				
A	B[b]	C[c]	After-flame (sec)	After-glow (sec)	Char Length (inch)	Char Length (cm)	Pass/Fail, Comments
Program Target			0	≤25	≤3.5	≤8.9	
Kevlar (41%)	FR polyester, WR finish (41%)	Binder (18%)	0	0	1.0	2.5	Fail; flash over entire surface.
Kynol (41%)	FR polyester, WR finish (41%)	Binder (18%)	0	0	2.4	6.1	Pass
P84, 1.5 den (1.7 dtex), WR finish (41%)	FR polyester, WR finish (41%)	Binder (18%)	0	0	2.5	6.4	Tentative Pass; 4-inch (10 cm) long surface flash
P84, 0.55 den (0.61 dtex), WR finish (82%)	None	Binder (18%)	0	0	0.7	1.8	Pass; batt shrinks away from ignition source
Pyroloft® manufactured by Albany/Lenzing Venture; essentially the same as the P84, 0.55 den (0.61 dtex) blend immediately above.			0	0	0.9	2.3	Pass; batt shrinks away from ignition source

a. All samples were 4 oz/yd^2 (135 g/m^2) bonded batts of 0.67 inch (1.70 cm) thickness (nominal; variation existed) with a nominal volume density of 0.50 lb/ft^3 (8.0 kg/m^3). All tests were machine direction (MD) tests; i.e., the fold was perpendicular to the MD. One test was made per sample type.

b. All FR polyester cited in this column, with WR (Si water repellent), was 1.5 denier (1.7 dtex) product supplied by Hoechst Celanese.

c. The binder fiber used in all samples was Hoechst Celanese Type K54, 4 denier (4.4 dtex).

Table 10. ***Results of Horizontal, Flat Batt, Methenamine Pill, Flammability Tests (Variation of Fed. Std. 191A, Method 5907) Made on a Series of Multicomponent Batts Prepared Using a 12-inch (30-cm) Machine Card***

Fiber Components[a]			Flammability Test Results				
			After-flame (sec)	After-glow (sec)	Char Length		Pass/Fail, Comments
A	B[b]	C[c]			(inch)	(cm)	
Program Target			0	≤25	≤3.5	≤8.9	
Kevlar (41%)	FR polyester, WR finish (41%)	Binder (18%)	0	0	0.5	1.3	Fail; flash over much of surface.
Kynol (41%)	FR polyester, WR finish (41%)	Binder (18%)	0 0 0 0	0 0 0 0	2.0 >3.2 >1.6 1.8	5.1 >8.1 >4.1 4.6	Tentative Pass; propagation of char may have been limited by small sample size.
P84, 1.5 den (1.7 dtex), WR finish (41%)	FR polyester, WR finish (41%)	Binder (18%)	0 0 0 0 0	0 0 0 0 0	1.6 2.4 >3.5 1.6 >3.0	4.1 6.1 >8.9 4.1 >7.6	Tentative Pass; propagation of char may have been limited by small sample size.
P84, 0.55 den (0.61 dtex), WR finish (82%)	None	Binder (18%)	0 0 0 0 0	0 0 0 0 0	1.5 1.2 1.2 1.4 1.2	3.8 3.0 3.0 3.6 3.0	Pass; no flash
Pyroloft® manufactured by Albany/Lenzing Venture; essentially the same as the P84, 0.55 den (0.61 dtex) blend immediately above.			0 0 0 0 0	0 0 0 0 0	1.1 0.9 1.2 1.1 1.2	2.8 2.3 3.0 2.8 3.0	Pass; very minor, local flash

a. All samples were 4 oz/yd^2 (135 g/m^2) bonded batts of 0.67 inch (1.70 cm) thickness (nominal; variation existed) with a nominal volume density of 0.50 lb/ft^3 (8.0 kg/m^3). Only one 9 x 9 inch (23 x 23 cm) piece of each sample type was available, and so, in all instances not limited by char length or flash, five tests were made on each piece.

b. All FR polyester cited in this column, with WR (SI water repellent), was 1.5 denier (1.7 dtex) product supplied by Hoechst Celanese.

c. The binder fiber used in all samples was Hoechst Celanese Type K54, 4 denier (4.4 dtex).

Table 11 E. ***Thermal Conductivity of Four Multicomponent Test Batts Prepared Using a 12-inch Machine Card***

Fiber Components			Batt Thickness (inch)	Batt Density (lb/ft^3)	Mean Temp. (°F)	Apparent Thermal Conductivity[b] ($Btu\text{-}in/hr\text{-}ft^2\text{-}°F$)
A	B	C[a]				
Kevlar (41%)	Polyester, FR, WR, 1.5 den (41%)	Binder (18%)	1.84	0.50	75	0.301
Kynol (41%)	Polyester, FR, WR, 1.5 den (41%)	Binder (18%)	2.15	0.56	74	0.297
P84, WR, 1.5 den (41%)	Polyester, FR, WR, 1.5 den (41%)	Binder (18%)	1.80	0.52	75	0.290
P84, WR, 0.55 den (82%)[c]	None	Binder (18%)	1.93	0.51	74	0.252

a. The binder used in all samples was Hoechst Celanese Type K54, 4 denier.

b. The program target was ≤0.300 $Btu\text{-}in/hr\text{-}ft^2\text{-}°F$.

c. Pyroloft® manufactured by Albany International.

- All tests were made in accord with the plate/sample/plate method described in ASTM C518. Heat flow was downward; T_1 = 100°F; T_2 = 50°F. A 0.5 oz/yd^2 nonwoven scrim was used on the top and bottom of all samples.

Table 11 (SI). *Thermal Conductivity of Four Multicomponent Test Batts Prepared Using a 30-cm Machine Card*

Fiber Components			Batt Thickness (cm)	Batt Density (kg/m^3)	Mean Temp. (°C)	Apparent Thermal Conductivity[b] (W/m-K)
A	B	C[a]				
Kevlar (41%)	Polyester, FR, WR, 1.7 dtex (41%)	Binder (18%)	4.67	8.0	24	0.0433
Kynol (41%)	Polyester, FR, WR, 1.7 dtex (41%)	Binder (18%)	5.46	9.0	23	0.0428
P84, WR, 1.7 dtex (41%)	Polyester, FR, WR, 1.7 dtex (41%)	Binder (18%)	4.57	8.3	24	0.0418
P84, WR, 0.61 dtex (82%)[c]	None	Binder (18%)	4.90	8.2	23	0.0363

a. The binder used in all samples was Hoechst Celanese Type K54, 4.4 dtex.

b. The program target was ≤0.0432 W/m-K.

c. Pyroloft® manufactured by Albany International.

- All tests were made in accord with the plate/sample/plate method described in ASTM C518. Heat flow was downward; T_1 = 38°C; T_2 = 10°C. A 17 g/m^2 nonwoven scrim was used on the top and bottom of all samples.

5. The water absorptive capacity data of Tables 12 and 13 provided assurance that the absorptive capacity target of ≤ 150% after 20 minutes of immersion could be met. It also showed that further fiber finish work would be required if certain blend combinations were selected. An important consideration regarding absorptive capacity which is not evident in Tables 12 and 13 is the contribution that microfiber components make toward reducing the absorptive capacity of low density batts.

6. The P84 microfiber (0.55 denier) used in the test series performed well in terms of flame resistance, insulating capacity and water repellency, confirming the adequacy of the manufacturer's finish application and making the fiber a very strong candidate.

D. Five Final Candidate Blends; Selection of One

The observations and conclusions reported above, prior experience, and an assessment of relative materials costs were the basis for selection of the next set of experimental blends. It was anticipated that the set might include a blend that was satisfactory in performance terms and so fiber cost was factored into the selection process to ensure the practicality of the final candidate. Five blends were chosen and all contained P84 microfiber, in weight fractions ranging from 22 to 60%, as the primary performance fiber. Each of the blends contained a different secondary fiber that was chosen in an attempt to mitigate the relatively high cost of P84 microfiber without reducing performance below target values. Hoechst Celanese K54 binder fiber was used in all of the blends at a weight fraction of 18%; binder fiber variations were to be considered later. The composition and the materials cost (based upon fiber costs at program outset) of the five sample batt types were as follows:

1. P84, 0.55 denier, WR (60%) / Wellman polyester, 0.5 denier, non-FR, WR (22%) / K54 binder fiber (18%); $15.11/lb

2. P84, 0.55 denier, WR (60%) / Hoechst Celanese polyester, 1.5 denier, FR, WR (22%) / K54 binder (18%); $15.05/lb

3. P84, 0.55 denier, WR (41%) / Kynol, 2 denier, non-WR (41%) / K54 binder (18%); $13.39/lb

Table 12. ***Water Absorptive Capacity (%)[a] of Multicomponent Batts After 20 Minutes of Immersion***

Sample No.	Kevlar (41%) Blend	Kynol (41%) Blend[b]	P84 Macrofiber (41%) Blend[b]	P84 Microfiber (82%) Blend[c]	Pyroloft®, AI[d]	WR Kevlar (82%) Blend[e]	WR Kynol (82%) Blend[e]
1	1770	1442	856	142	166	404	123
2	1497	1155	1345	131	252	517	140
3	1561	1411	1287	139	215	233	127
4	1613	1391	1104	136	165		
5	1611	1230	579	157	200		
6	1405		1183				
7			699				
8			1031				
Average	1576	1326	1011	141	200	385	130

a. Test Method based on ASTM D117 as modified in Natick Technical Report TR-891021.

b. Made using 12-inch (30-cm) machine card. Other components in the three-component blend are: Hoechst Celanese polyester, 1.5 denier (1.7 dtex), FR, WR (41%) and Hoechst Celanese binder, 4 denier (4.4 dtex) [18%].

c. Made using 12-inch (30 cm) machine card. The other component in the two-component blend is: Hoechst Celanese binder, 4 denier (4.4 dtex) [18%].

d. Essentially the same as the blend referenced in c., above, but manufactured by Albany International.

e. Made using Spinlab table-top opener/blender. The other component in the two-component blend is: Hoechst Celanese binder, 4 denier (4.4 dtex) [18%]. The WR treatment on Kevlar and Kynol is Zonyl fluorocarbon, applied at AI Research.

Table 13. *Water Absorptive Capacity (%)[a] of Opened Fiber After 20 Minutes of Immersion*

Sample No.	Hoechst Celanese Polyester with WR	Hoechst Celanese Polyester w/o WR, Scoured[b]	Hoechst Celanese Polyester w/o WR, Scoured, Zonyl Treated[c]	Kevlar	Kevlar, Scoured[b]	Kevlar Scoured, Zonyl Treated[c]	Kynol	Kynol, Scoured[b]	Kynol Scoured, Zonyl Treated[c]	P84, 0.55 den (0.61 dtex), Excess WR Finish	P84, 0.55 den (0.61 dtex), Standard WR Finish	P84, 1.5 den (1.7 dtex), Excess WR
1	711	1591	228	1723	1581	255	814	1089	141	1787	209	1593
2	729	1551	315	1567	1522	370	817	1074	185	1498	148	1743
3	327	1757	189	1523	1571	275	825	977	140	1661	217	1600
4	464	1596	224	1567	1707	309	801	931	128	1563	251	1492
5	355	1538	168	1619	1609	382	760	925	144	1705	161	1699
6	402	1712	495	1621	1867	599	770	1047	146	1679	461	1877
7	600	1454	233	1705	1549	493	862	1148	230	1591	224	1787
8	339	1401	247	1501	1653	348	849	1072	154	1638	179	1731
9	217	1314	182	1646	1432	325	852	1063	185	1634	211	1727
10	252	1592	164	1538	1555	154	855	1021	163	1572	281	1735
Average	567	1551	245	1601	1605	351	821	1035	162	1633	234	1698

a. Test Method based on ASTM D1117 as modified in Natick Technical Report TR-86102l.
b. Scoured with 1% Merpol HCS for 30 minutes at 170°F (77°C) and dried at 200°F (93°C).
c. Scoured with 1% Merpol HCS for 30 minutes at 170°F (77°C) and dried at 200°F (93°C), then treated with Ciba Zonyl fluorocarbon (1.3 g/l) and dried at 320°F (160°C).

4. P84, 0.55 denier, WR (41% / Kevlar, 1.5 denier, non-WR (41%) / K54 binder fiber (18%); $14.82/lb
5. P84, 0.55 denier, WR (22%) / P84, 1.5 denier, WR (60%) / K54 binder (18%); $14.55/lb

The materials costs shown for the sample set range from $13.39/lb to $15.11/lb, comparing favorably to the $10-12/lb cost of aramid fiber currently used in a Mil Spec quilted batting for cold weather clothing. It was anticipated that improvement in insulating value/unit weight over the current material would off-set the small materials-cost differential of any of the five candidates.

Bonded batt samples were made with each of the five P84 microfiber blends, using a 12-inch machine card, and the samples were subjected to three types of flammability tests and water absorption tests, as reported in Tables 14 through 17. Review of the new data and application of the cumulative experience of the program led to a series of observations, conclusions and decisions that became the bases for selection of the optimal staple blend, as follows:

1. The first two fiber/batt configurations listed in Table 14 (vertical flammability test results) contained, by weight, 22% polyester (exclusive of the polyester binder component common to all configurations). In one case, the polyester included a fire retardant, phosphorus additive; in the other case it did not. The relatively low fraction of polyester in each blend had been chosen as approximately the maximum fraction that might allow a blended-fiber batt to exhibit acceptable vertical flammability performance. It was also reasoned that inclusion of some polyester to reduce net materials cost was highly desirable, but that a weight fraction less than about 20% would be of greatly diminished practicality. However, these two further attempts to utilize polyester became the final ones. The data of Table 14 shows that, in both cases, the char lengths measured after vertical flammability tests greatly exceeded the program target. This led to a decision to eliminate polyester (except for the necessary binder fiber component) from further consideration.

2. The vertical flammability test results of Table 14 were the basis for elimination of another constituent-fiber candidate, Kevlar. The fourth-listed blended-fiber batt candidate consisted of 0.55 denier P84 (41%), Kevlar (41%) and binder fiber (18%) and had been selected with several objectives in mind. Surface flash had been a persistent phenomenon with most Kevlar-containing batts tested during the program. In previous attempts to utilize Kevlar, it was the predominant blend fiber, by weight, but in this Kevlar-utilization effort, P84 microfiber and Kevlar were blended on an equal weight basis. It was reasoned that such a blend might: (a) minimize Kevlar's tendency to flash, (b) utilize the insulating advantage of P84 microfiber, and (c) be materials-cost effective. The

Table 14. ***Results of Vertical Flammability Tests[a] Made on a Series of Three-Component Batts Prepared Using a 12-inch (30-cm) Machine Card***

Fiber Components: A	Fiber Components: B	Fiber Components: C[b]	Test Direction	After-flame (sec)	After-glow (sec)	Char Length (inch)	Char Length (cm)	Pass/Fail, Comments
Program Target			-	0	≤25	≤3.5	≤8.9	
P84, 0.55 den (0.61 dtex), WR finish (60%)	Wellman polyester, 0.5 den (0.55 dtex), non-FR, WR finish (22%)	Binder (18%)	MD	6	0	3.0	7.6	
			MD	27	0	9.0	23	
			MD	31	0	9.0	23	
			MD	22	0	9.0	23	
			MD	33	0	9.0	23	
			MD, Avg.	24	0	7.8	20	
			XD	40	0	9.0	23	
			XD	40	0	9.0	23	
			XD	37	0	9.0	23	
			XD	31	0	9.0	23	
			XD	44	0	9.0	23	
			XD, Avg.	38	0	9.0	23	Fail
P84, 0.55 den (0.61 dtex), WR finish (60%)	Hoechst Celanese polyester, 1.5 den (1.7 dtex), FR, WR finish (22%)	Binder (18%)	MD	40	0	9.0	23	
			MD	31	0	9.0	23	
			MD	33	0	9.0	23	
			MD	12	0	5.0	13	
			MD	4	0	4.0	10	
			MD, Avg.	24	0	7.2	18	
			XD	35	0	9.0	23	
			XD	32	0	9.0	23	
			XD	47	0	9.0	23	
			XD	35	0	9.0	23	
			XD	24	0	9.0	23	
			XD, Avg.	35	0	9.0	23	Fail
P84, 0.55 den (0.61 dtex), WR finish (41%)	Kynol, 2 den (2.2 dtex), non-WR (41%)	Binder (18%)	MD	0	2	0.8	2.0	
			MD	0	2	1.0	2.5	
			MD	0	2	1.0	2.5	
			MD	0	2	1.0	2.5	
			MD	0	2	1.5	3.8	• Minor surface flash on 3 XD samples
			MD, Avg.	0	2	1.1	2.8	
			XD	0	1	1.0	2.5	
			XD	0	1	1.5	3.8	• Reported char lengths include shrinkage[c]
			XD	0	1	1.5	3.8	
			XD	0	2	1.5	3.8	
			XD	0	2	1.5	3.8	
			XD, Avg.	0	1	1.4	3.6	• Pass

a. Test method and sample configuration as given in Table 5, footnote a.
b. The binder fiber used in all samples was: Hoechst Celanese Type K54, 4 denier (4.4 dtex).
c. Shrinkage typically comprised about 0.5 inch (1.3 cm) of these char lengths.

Note: ***Table continues on next page.***

Table 14. ***Results of Vertical Flammability Tests[a] Made on a Series of Three-Component Batts Prepared Using a 12-inch (30-cm) Machine Card (continued)***

Fiber Components			Flammability Test Results					Pass/Fail, Comments
A	B	C[b]	Test Direction	After-flame (sec)	After-glow (sec)	Char Length (inch)	Char Length (cm)	
Program Target			–	0	≤25	≤3.5	≤8.9	
P84, 0.55 den (0.61 dtex), WR finish (41%)	Kevlar, 1.5 den (1.7 dtex), non-WR (41%)	Binder (18%)	MD	0	1	0.5	1.3	• Major surface flash on 2 MD samples
			MD	0	1	1.2	3.0	
			MD	0	1	1.2	3.0	
			MD	0	1	0.8	2.0	
			MD	0	1	0.8	2.0	
			MD, Avg.	0	1	0.9	2.3	• Reported char lengths include shrinkage[c]
			XD	0	1	0.8	2.0	
			XD	0	1	0.8	2.0	
			XD	0	1	0.4	1.0	• Pass; tentative only, due to tendency toward major surface flash
			XD	0	1	0.4	1.0	
			XD	0	1	0.4	1.0	
			XD, Avg.	0	1	0.6	1.5	
P84, 0.55 den (0.61 dtex), WR finish (22%)	P84, 1.5 den, (1.7 dtex), WR finish (60%)	Binder (18%)	MD	0	2	1.2	3.0	
			MD	0	2	1.2	3.0	
			MD	0	3	1.2	3.0	
			MD	0	4	1.2	3.0	
			MD	0	4	1.2	3.0	
			MD, Avg.	0	3	1.2	3.0	
			XD	0	5	1.2	3.0	
			XD	0	5	1.0	2.5	• No surface flash in any of 10 tests
			XD	0	5	1.2	3.0	
			XD	0	8	1.2	3.0	
			XD	0	5	1.2	3.0	• Reported char lengths include shrinkage[c]
			XD, Avg.	0	6	1.2	3.0	

a. Test method and sample configuration as given in Table 5, footnote a.
b. The binder fiber used in all samples was: Hoechst Celanese Type K54, 4 denier (4.4 dtex).
c. Shrinkage typically comprised about 0.5 inch (1.3 cm) of these char lengths.

above-average cost of P84 fiber, in comparison to other viable, inherently FR fiber candidates, would be off-set by Kevlar's slightly below-average cost. However, major surface flash was observed on two of the ten Kevlar-containing vertical flammability samples; prior experience had shown that this is adequate indication of the undesirable characteristic. Although surface flash and its overall significance are not fully understood, it is a decidedly negative phenomenon that can be avoided; eliminating Kevlar fiber from further consideration largely eliminated concern regarding surface flash.

3. The fiber blend that is listed third in Table 14 had been selected for evaluation through reasoning similar to that described above for the equal-weight P84 microfiber / Kevlar blend. This third-listed blend contained 0.55 denier P84 (41%), Kynol (41%) and binder fiber (18%) and represented an attempt to: (a) minimize previously observed flammability shortcomings of Kynol, including a tendency to surface flash, (b) achieve acceptable insulating performance by combining a 0.55 denier microfiber (P84) with a relatively large-diameter fiber, 2 denier Kynol, and (3) balance P84 microfiber's above-average cost with Kynol's below-average cost. The equal-weight P84 microfiber/Kynol blend exhibited flammability resistance well within program targets (Tables 14, 15, and 16), but some surface flash was observed on three of ten vertical flammability test specimens. The surface flashes seen were minor in comparison to those observed on the Kevlar blend specimens discussed above, but, nonetheless, were regarded as a negative aspect. Program experience had shown that the flash phenomenon is highly dependent upon the degree of hairiness of the batt surface, and hairiness inevitably varies among batts of the same blend made under nominally equivalent, but subtly different, conditions. Consequently, we remained wary of recommending any blend that had shown a tendency to flash. Further disadvantages existed in relation to utilizing Kynol in a blend of the type shown in Table 14, as follows: (a) its fiber diameter is relatively high (on the order of 14 microns), making it undesirable in terms of insulating efficiency, and (b) it is not commercially available with a water repellent finish.

4. The fifth and final blended batt listed in Table 14 consisted of: 0.55 denier P84, with WR (22%), 1.5 denier P84, with WR (60%) and binder fiber (18%). This blend was selected, at this point in the effort, as the final candidate. The selection was based upon: (a) wholly acceptable flammability test results (Tables 14, 15, and 16), without any tendency toward surface flash, (b) acceptable water absorption capacity data (Table 17), a consequence of the fluorocarbon, water repellent finish applied by the fiber manufacturer, Lenzing, (c) a high level of confidence that the thermal conductivity target and all other program targets would be met by this configuration, (d) a reasonable

Table 15. ***Results of Folded Batt, Methenamine Pill, Flammability Tests (Variation of Fed. Std. 191A, Method 5907) Made on a Series of Multicomponent Batts Prepared Using a 12-inch (30-cm) Machine Card***

Fiber Components[a]			Flammability Test Results						Pass/Fail Comments
A	B	C[b]	After-flame (sec)	After-glow (sec)	Char Length[c] (inch)	Char Length[c] (cm)	Flash Length[d] (inch)	Flash Length[d] (cm)	
Program Target			0	≤25	≤3.5	≤8.9	–	–	
P84, 0.55 den (0.61 dtex), WR finish (60%)	Wellman polyester, 0.5 den (0.55 dtex), non-FR, WR finish (22%)	Binder (18%)	2.5	0	0.9	2.3	0	0	Fail
P84, 0.55 den (0.61 dtex), WR finish (60%)	H. Cel. polyester, 1.5 den (1.7 dtex), FR, WR finish (41%)	Binder (18%)	2.0	0	1.3	3.3	0	0	Fail
P84, 0.55 den (0.61 dtex), WR finish (41%)	Kynol, 2 den (2.2 dtex), non-WR (41%)	Binder (18%)	0	0	1.2	3.0	0	0	Pass; batt shrinks away from ignition source
P84, 0.55 den (0.61 dtex), WR finish (41%)	Kevlar, 1.5 den (1.7 dtex), non-WR (41%)	Binder (18%)	0	0	1.3	3.3	0	0	Pass; batt shrinks away from ignition source
P84, 0.55 den (0.61 dtex), WR finish (22%)	P84, 1.5 den (1.7 dtex), WR finish (60%)	Binder (18%)	0	0	0.9	2.3	0	0	Pass; batt shrinks away from ignition source

a. All samples were 4 oz/yd^2 (135 g/m^2) bonded batts of 0.67 inch (1.70 cm) thickness (nominal; variation existed) with a nominal volume density of 0.50 lb/ft^3 (8.0 kg/m^3). All tests were machine direction (MD) tests; i.e., the fold was perpendicular to the MD. One test was made per sample type.

b. The binder used in all samples was: Hoechst Celanese Type K54, 4 denier (4.4 dtex).

c. Char Length: The longest length of material which has been charred through more than two dimensions of the sample.

d. Flash Length: The longest dimension of the area of discoloration/degradation of the material due to exposure to flame and/or heat, usually only in two dimensions (i.e., not through the thickness of the material; a surface effect).

Table 16. ***Results of Horizontal, Flat Batt, Methenamine Pill, Flammability Tests (Variation of Fed. Std. 191A, Method 5907) Made on a Series of Multicomponent Batts Prepared Using a 12-inch (30-cm) Machine Card***

Fiber Components[a]			Flammability Test Results						
A	B	C[b]	After-flame (sec)	After-glow (sec)	Char Length[c] (inch)	Char Length[c] (cm)	Flash Length[d] (inch)	Flash Length[d] (cm)	Pass/Fail Comments
Program Target			0	≤25	≤3.5	≤8.9	–	–	
P84, 0.55 den (0.61 dtex), WR finish (60%)	Wellman polyester, 0.5 den (0.55 dtex), non-FR, WR finish (22%)	Binder (18%)	0 0 0 0 0	0 0 0 0 0	0.9 1.4 1.3 0.8 1.2	2.3 3.6 3.3 2.0 3.0	1.8 0 0 1.6 0	4.6 0 0 4.1 0	Tentative pass; limited surface flash
P84, 0.55 den (0.61 dtex), WR finish (60%)	H. Cel. polyester, 1.5 den (1.7 dtex), FR, WR finish (22%)	Binder (18%)	0 0 0 0 0	0 0 0 0 0	0.8 0.8 0.9 0.8 0.8	2.0 2.0 2.3 2.0 2.0	2.2 >1.8 1.6 1.3 >2.2	5.6 >4.6 4.1 3.3 >5.6	Tentative pass; surface flash with limited flame propagation
P84, 0.55 den (0.61 dtex), WR finish (41%)	Kynol, 2 den (2.2 dtex), non-WR (41%)	Binder (18%)	0 0 0 0 0	0 0 0 0 0	1.4 1.4 1.4 1.3 >1.5	3.6 3.6 3.6 3.3 >3.8	0 0 0 0 0	0 0 0 0 0	Pass; no propagation; char due to direct flame exposure
P84, 0.55 den (0.61 dtex), WR finish (41%)	Kevlar, 1.5 den (1.7 dtex), non-WR (41%)	Binder (18%)	0 0 0 0 0	0 0 0 0 0	0.6 0.8 1.0 0.8 0.9	1.5 2.0 2.5 2.0 2.3	1.2 1.4 1.6 1.2 1.4	3.0 3.6 4.1 3.0 3.6	Tentative pass; surface flash; no propagation; char due to direct flame exposure
P84, 0.55 den (0.61 dtex), WR finish (22%)	P84, 1.5 den (1.7 dtex), WR finish (60%)	Binder (18%)	0 0 0 0 0	0 0 0 0 0	1.2 1.2 1.0 1.0 1.3	3.0 3.0 2.5 2.5 3.3	0 0 0 0 0	0 0 0 0 0	Pass; no propagation; char due to direct flame exposure

a. All samples were 4 oz/yd^2 (135 g/m^2) bonded batts of 0.67 inch (1.70 cm) thickness (nominal; variation existed) with a nominal volume density of 0.50 lb/ft^3 (8.0 kg/m^3). Only one 9 x 9 inch (23 x 23 cm) piece of each sample type was available, and so, in all instances not limited by char length or flash, five tests were made on each piece.
b. The binder used in all samples was: Hoechst Celanese K54, 4 denier (4.4 dtex).
c. Char Length: The longest length of material which has been charred through more than two dimensions of the sample.
d. Flash Length: The longest dimension of the area of discoloration/degradation of the material due to exposure to flame and/or heat, usually only in two dimensions (i.e., not through the thickness of the material; a surface effect).

Table 17. ***Water Absorptive Capacity, After 20 Minutes Immersion, of Bonded Batt and Opened Fiber Specimens of Particular Interest***

Bonded Batt Specimens (1.5 g.dry)				
Fiber Components			After 20 Min. Immersion	
A	B	C	Wet Weight (g)	Absorptive Capacity (%)
Program Target			--	150
P84, 0.55 den (0.61 dtex), WR finish (41%)	Kynol, 2 den (2.2 dtex), non-WR (41%)	Binder, H. Cel. Type K54, 4 den (4.4 dtex) (18%)	3.66 2.73 2.62 $\bar{x}$ 3.00	244 182 175 $\bar{x}$ 200
P84, 0.55 den (0.61 dtex), WR finish (22%)	P84, 1.5 den (1.7 dtex), WR finish (60%)	Binder, H. Cel. Type K54, 4 den (4.4 dtex) (18%)	2.13 2.11 2.11 $\bar{x}$ 2.12	142 141 141 $\bar{x}$ 141
P84, 0.55 den (0.61 dtex), WR finish (22%)	P84, 1.5 den (1.7 dtex), WR finish (60%)	Binder, H. Cel. Type 255, 3 den (3.3 dtex) (18%)	2.16 1.81 2.01 $\bar{x}$ 1.99	144 121 134 $\bar{x}$ 133

Opened Fiber Specimens, Data Summary
Kynol, 2 den (2.2 dtex), non-WR; average absorptive capacity, 10 tests = 821%
P84, 0.55 den (0.61 dtex), WR; average absorptive capacity, 10 tests = 234%
P84, 1.5 den (1.7 dtex), WR; average absorptive capacity, 10 tests = 133%

materials cost of approximately $14.50/lb of insulator, due largely to the 60/22, P84 macrofiber / P84 microfiber ratio, which accommodates the relatively high cost of P84 microfiber, and (e) versatility provided by using all P84 (and binder fiber); later in the program, and/or after completion of the program, adjustments could readily be made to the P84 macrofiber/microfiber ratio to either reduce materials cost (at the expense of insulating performance) or to improve insulating performance (at higher cost). Experimental work reported previously herein provides assurance that such blend ratio adjustments would not adversely affect flammability resistance and other batt performance parameters. Virtually all other three-component blends resulted in batt flammability characteristics that were sensitive to blend ratio.

E. Evaluation of Alternative Binder Fibers

After the P84 microfiber/macrofiber blend was chosen, further attention was given to binder fiber choice. The binder component used in most experimental work prior to that point was Hoechst Celanese Type K54, a sheath/core, polyester/polyester, bicomponent fiber that is one in a series of similar binder fibers available from Hoechst Celanese. We had previously selected Type K54, after extended experimentation, for use in Primaloft and it has also been used successfully with P84 fiber in commercial insulator applications. Our prior experience, together with wholly satisfactory flammability test results for P84/Type K54 blends during the course of this program, had made Type K54 the leading, but tentative, binder fiber choice. Although it appeared that Type K54 would perform acceptably, an opportunity to improve laundering durability, through selection of a similar binder with a higher softening/melt temperature, seemed to exist. The first criterion set for alternative binders was that they must not have a greater negative effect upon flammability than Type K54. Three types were chosen for evaluation through the vertical flammability testing of each in a bonded, P84 macrofiber/microfiber batt. They were:

(1) Hoechst Celanese Type 255, polyester/polyolefin, 261°F melt temperature,

(2) Hoechst Celanese Type K53, polyester/polyester, 266°F melt temperature, and

(3) Hoechst Celanese Type 252, polyester/polyester, 392°F melt temperature.

The melt temperature for Type K54 is 230°F. This and the other melt temperatures given are reference values supplied by Hoechst Celanese; some softening and flow occurs at lower temperatures. All four binders (Type K54 and the three alternatives) are sheath/core types and those used in this work were of

3 or 4 denier, depending upon the availability of each, and were 1.5 inches in length. It had been determined, early in the work, that staple lengths greater than 1.5 inches were difficult to blend uniformly and that poorly blended polyester binder adversely affected vertical flammability test results. For this reason, it was decided that all fiber components in the final blend should be 1.5 inches in length.

Vertical flammability test results for bonded batts made using P84 microfiber/macrofiber (22% / 60%) and each of the four binder types (18%) are reported in Table 18. Data for all four compared favorably to program target values for afterflame, afterglow and char length, but important reservations existed regarding the behavior of each of the three higher-temperature alternatives to Type K54. The batt samples containing Type 255 binder exhibited unusual secondary surface flame in four of ten tests and the Type 252 and Type K53 samples were prone to much longer afterglow times than those we had become accustomed to observing. In fact, two of nine afterglow times reported for Type K53 samples exceeded the program target of ≤ 25 seconds, although the nine-specimen average did not. These results were taken as ample indication that none of the three binder alternatives being considered would offer the minimal flammability influence of Type K54 binder. Thus, our original binder selection received further, although somewhat indirect, substantiation and was not changed.

F. Summary

The experimental effort reported in this and the previous section began with the selection of ten credible FR fiber candidates and four binder fiber candidates and led to the selection of a fiber blend that could be processed to make a bonded, high loft, FR, insulating batt of reasonable cost and with a very high probability of meeting or exceeding all program performance objectives. This blend, as discussed above, consisted of the following fiber components:

(1) P84 polyimide microfiber; 0.55 denier x 1.5 inch; with fluorocarbon, WR finish applied by the fiber manufacturer, Lenzing; 22% of blend, by weight,

(2) P84 polyimide macrofiber; 1.5 denier x 1.5 inch; with fluorocarbon, WR finish applied by the fiber manufacturer, Lenzing; 60% of blend, and

(3) Hoechst Celanese K54 binder fiber; polyester/polyester, sheath/core; 4 denier x 1.5 inch; 18% of blend.

Pilot line production of an FR insulator prototype based upon this blend will be reported in Section V.

Table 18. *Vertical Flammability Test Results[a] for Four P84 Batts that Differ Only in Binder Type*

Fiber Components			Flammability Test Results					
						Char Length		
A	B	C	Test Direction	Afterflame (sec)	Afterglow (sec)	(inch)	(cm)	Comments
Program Target			-	0	≤25	≤3.5	≤8.9	
P84, 0.55 den (0.61 dtex), WR finish (22%)	P84, 1.5 den (1.7 dtex), WR finish (60%)	Binder, H. Cel. Type K54, polyester/ polyester, 230°F melt, 4 den (4.4 dtex) (18%)	MD	0	2	1.2	3.0	
			MD	0	2	1.2	3.0	
			MD	0	3	1.2	3.0	
			MD	0	4	1.2	3.0	
			MD	0	4	1.2	3.0	
			MD, Avg.	0	3	1.2	3.0	
			XD	0	5	1.2	3.0	
			XD	0	5	1.0	2.5	
			XD	0	5	1.2	3.0	• No surface flash in any of 10 tests
			XD	0	8	1.2	3.0	
			XD	0	5	1.2	3.0	
			XD, Avg.	0	6	1.2	3.0	• Reported char lengths include shrinkage[b]
P84, 0.55 den (0.61 dtex), WR finish (22%)	P84, 1.5 den (1.7 dtex), WR finish (60%)	Binder, H. Cel. Type 255, polyester/ polyolefin, 261°F melt, 3 den (3.3 dtex) (18%)	MD	0	2	1.2	3.0	
			MD	0	2	1.2	3.0	
			MD	0	2	1.5	3.8	
			MD	0	2	1.2	3.0	
			MD	0	2	3.0	7.6	
			MD, Avg.	0	2	1.6	4.1	• Secondary (lesser) surface flame occurred in 4 of 10 tests, resulting in secondary char of aproximately double the length reported
			XD	0	2	1.8	4.6	
			XD	0	2	1.2	3.0	
			XD	0	2	1.5	3.8	
			XD	0	2	1.8	4.6	
			XD	0	1	2.0	5.1	
			XD, Avg.	0	2	1.7	4.3	• Reported char lengths include shrinkage[c]
P84, 0.55 den (0.61 dtex), WR finish (22%)	P84, 1.5 den (1.7 dtex), WR finish (60%)	Binder, H. Cel. Type K53, polyester/ polyester, 266°F melt, 4 den (4.4 dtex) (18%)	MD	0	2	0.8	2.0	
			MD	0	6	0.8	2.0	
			MD	0	2	0.8	2.0	
			MD	0	5	0.9	2.3	
			MD, Avg.	0	4	0.8	2.0	
			XD	0	5	0.5	1.3	• No surface flash in any of 9 tests
			XD	0	19	0.6	1.5	
			XD	0	8	0.5	1.3	• Reservations regarding long afterglow times
			XD	0	31	0.9	2.3	
			XD	0	28	0.9	2.3	
			XD, Avg.	0	10	0.7	1.8	• Reported char lengths include shrinkage[d]
P84, 0.55 den (0.61 dtex), WR finish (22%)	P84 1.5 den (1.7 dtex), WR finish (60%)	Binder, H. Cel. Type 252, polyester/ polyester, 392°F melt, 3 den (3.3 dtex) (18%)	MD	0	8	1.2	3.0	
			MD	0	6	1.0	2.5	
			MD	0	7	1.2	3.0	
			MD	0	6	0.8	2.0	
			MD	0	6	1.2	3.0	
			MD. Avg.	0	7	1.1	2.8	• No surface flash in any of 10 tests
			XD	0	10	0.8	2.0	• Reservations regarding afterglow times greater than those measured for similar blends
			XD	0	16	0.8	2.0	
			XD	0	11	0.8	2.0	
			XD	0	13	1.2	3.0	
			XD	0	15	1.2	3.0	
			XD, Avg.	0	13	1.0	2.5	• Reported char lengths include shrinkage[b]

a. Test method and sample configuration as given in Table 5, footnote a.
b. Shrinkage typically comprised about 0.5 inch (1.3 cm) of the char length.
c. Shrinkage component ranged from 0.5 to 2.5 inches (1.3 to 6.4 cm); shrinkage values in the 0.5-0.75 inch (1.3 to 1.9 cm) range were most common.
d. Shrinkage component ranged from 0.25 to 0.5 inch (0.6 to 1.3 cm).

4. CONTINUOUS FILAMENT INSULATOR DEVELOPMENT

A. Introduction

Producing continuous filament tow that is suitable for opening, spreading and cross-lapping (batt making)is a technology that, with minor exception, is practiced within the United States only by Hoechst Celanese Corporation. Most tow opening and spreading equipment in the United States is owned by Hoechst Celanese and leased to regular purchasers of their specially prepared tow. Consequently, this program's objective to develop flame-resistant, high efficiency thermal insulation prototypes, based upon both staple fiber and continuous filament, made a cooperative effort between Albany International Research and Hoechst Celanese a logical and promising arrangement. Prior to the start of the program, Hoechst Celanese agreed to work with us as a subcontractor in the continuous filament portion of the effort. In addition to their singular position of leadership in continuous filament tow and batt-making, Hoechst Celanese offered the following strengths to the program as it was being planned:

(1) They had cooperated similarly, although not on a formal subcontracting basis, in a previous Natick Center/AI Research high loft insulation program[3].

(2) They had experience with selection and application of water repellent finishes to spreadable tow.

(3) They had successfully adapted a phosphorus-based fire-retardant for use in polyester staple fiber, were willing to apply that experience to the making of FR polyester tow and were confident that such tow would meet the program's FR target values.

(4) They had plans to utilize their bicomponent binder fiber technology, which is the basis of a staple product line of great utility, in two spreading applications such as the one planned for this program.

The technical assets and experience which Hoechst Celanese were prepared to contribute to the continuous filament insulator portion of the program provided confidence, at the outset, that a worthwhile, flame-resistant, spread tow insulator would be developed.

In spite of Hoechst Celanese' role as a subcontractor, we had planned to evaluate any FR, spreadable, continuous filament tow that could be obtained. However, early in the work we learned that only one other fiber producer, Phillips Fibers (now Amoco) was prepared to provide FR tow. As was reported in Section II, *Evaluation of Flame-Resistant Fiber Candidates for Use in Staple and Continuous Filament Insulators*, Phillips Fibers made an experimental Ryton tow for the program, but it could not be opened and spread and they could not allocate the additional resources necessary to develop a useable tow. Also reported in Section II was our finding that Hoechst Celanese' FR polyester, in the form of a staple batt of the appropriate density (0.5 lb/ft^3), would not meet the program's flammability target values. After consultation with Al Research and Hoechst Celanese, Natick Center acknowledged that Hoechst Celanese' proposed FR polyester tow provided the only practical option for development of a high performance, FR, continuous filament insulator and revised the program's FR targets downward for the continuous filament insulator only (these changes were reported in Section II, page 13).

B. Continuous Filament Insulator Development; Challenges and Approach

Acceptance of Hoechst Celanese' proposed FR polyester tow as the only viable fiber candidate appeared to be a turning point, but it did not, in fact, result in a tractable development task. Difficult insulator and processing problems remained and, in several cases, the experimentation required to resolve them could only be done using high speed, production equipment. The issues requiring resolution fell within two primary categories, as follows:

1. <u>Fiber finish</u> was required to achieve the program's water repellency objectives and finish optimization demanded consideration of several interactive factors such as tow processability, batt bonding and batt flammability.

2. A <u>batt bonding</u> approach that would not significantly reduce FR performance, water repellency or loft remained to be developed.

Hoechst Celanese, as owner and/or practitioner of all of the relevant technology, was to take the leading development role for the continuous filament insulator. Al Research Co.'s role was: (1) to be a partner in planning, especially of the experimental trials, (2) to evaluate experimental samples and (3) to interpret interim and final results in relation to program objectives.

C. Fiber Finish

Identification of an appropriate fiber finish, finish level, and finishing method was essential to the successful development of an FR, continuous filament insulator. At the outset, Hoechst Celanese and Al Research staff members agreed that curable polydimethylsiloxane (silicone), suitably applied, was an almost inevitable finish choice. The advantages known or anticipated for polydimethylsiloxane were these:

1. Fiber-to-fiber and fiber-to-metal friction is a major factor in tow opening and spreading and Hoechst Celanese has, for many years, relied upon silicone to provide a unique combination of "tack" and lubricity.

2. Al Research experience and that of at least two fiber producers (neither being Hoechst Celanese) has shown that curable polydimethylsiloxane fiber finish can impart the desired level of water repellency to low density batting made of small diameter polyester fiber.

3. Al Research has had success in using bicomponent, thermoplastic binder fiber to bond silicone treated fiber, in spite of its lubricity.

4. Although preliminary work showed that silicone finish had a measurable negative effect upon batt flammability resistance, it was perceived to be relatively minor.

Over a period of approximately one year, Hoechst Celanese expended considerable effort in laboratory and production line experimentation with fiber finish, most of it directed toward obtaining an acceptable level of water repellency with polydimethylsiloxane. During the period, Al Research evaluated the results of Hoechst Celanese' major finishing trials, using the water absorptive capacity test specified in the contract work statement. The results of these absorptive capacity tests are summarized in Table 19, primarily to show the scope of the effort made. None of the results compare favorably to the program target for water absorptive capacity of $\leq 150\%$. After each of the trials for which data is shown, and after many other less formal laboratory experiments, we conferred with Hoechst Celanese staff members. We frequently discussed: (1) suspected interference by hydrophilic spin finishes or other spinning-related contaminants, (2) suspected non-uniformity of application, (3) Al Research's success and that of other fiber producers in applying nominally-equivalent finishes, and (4) the existence of competing manufacturers of silicone finishes and the technical assistance they provide.

Table 19. *Summary of Water Absorptive Capacity Test Results Obtained at AI Research in Support of Hoechst Celanese Fiber Finishing Trials*

Fiber Sample[a] Description	Finish Description	Scoured and/or Finished By	Date	No. of Test Samples	Average[b] Absorptive Capacity (%)
FR polyester staple, 1.5 den (1.7 dtex)	Polydimethylsiloxane, 0.4% add-on	H. Cel.	Oct.' 92	20	506
FR polyester staple, 1.5 den (1.7 dtex)	Scoured in 1% Merpol solution	AI	Oct. '92	10	1550
FR polyester staple, 1.5 den (1.7 dtex)	Scoured and treated with Zonyl fluorocarbon	AI	Nov. '92	10	245
FR polyester staple, 1.5 den (1.7 dtex)	Polydimethylsiloxane, 0.4% add-on, "more uniform" than Oct. '92	H. Cel.	Dec. '92	20	967
FR polyester staple, 1.5 den (1.7 dtex)	Polydimethylsiloxane, 0.6% add-on	H. Cel.	Dec. '92	20	880
FR polyester staple, 1.5 den (1.7 dtex)	Ethylene oxide/propylene oxide chain, 0.4%	H. Cel.	Dec. '92	10	1148
FR polyester staple, 1.2 den (1.3 dtex)	Scoured and treated, polydimethylsiloxane, 0.4% add-on	H. Cel.	Feb. '93	10	648
FR polyester staple, 1.5 den (1.7 dtex)	Scoured and treated, polydimethylsiloxane, 0.4% add-on	H. Cel.	Feb. '93	10	585
Non-FR polyester, c.fil., 5 den (5.6 dtex)	Polydimethylsiloxane, 0.4% add-on	H. Cel.	Sept. '93	3	782
FR polyester, c.fil., 1.2 den (1.3 dtex)	"Improved" polydimethylsiloxane, 0.4% add-on	H. Cel.	Oct. '93	5	699

a. Although identifying a suitable continuous filament finish was the objective, many samples were cut into staple to simplify laboratory work.

b. The program target was ≤ 150%.

The inability of the only producer of spreadable, FR, continuous filament tow to apply a satisfactory water repellent finish to tow constituted a major impediment to program progress and one over which we had little control.

Prior to completion of the finish application trials for which data is shown in Table 19, Hoechst Celanese suggested, and we agreed, that FR polyester tow should be made for opening and spreading trials to address other development issues. Most of the remaining issues, as mentioned previously, related to batt bonding.

D. Continuous Filament Insulator Bonding

While planning the program with Hoechst Celanese, it was readily agreed that the acrylic resin binder that they had applied as a two-surface spray to most previously produced continuous filament insulators would not be acceptable. This binder system was seen as a liability in terms of flame resistance and water repellency. Instead, Hoechst Celanese proposed that they adapt their bicomponent, staple, binder fiber technology to the continuous filament batt-making process.

Early in the program, Hoechst Celanese investigated eight approaches to incorporating bicomponent binder fiber, in either continuous filament or staple form, in a high loft, continuous filament batting. The fiber configuration in each of the eight variations tried was as follows:

1. Batt made entirely of Type 254 binder (50/50, sheath/core) in continuous filament form.

2. Batt made of alternating layers of Type 254 binder and 5 denier polyester; both continuous filament; 50/50 blend.

3. Batt made from a single tow bundle that had been prepared by mixing bands of Type 254 binder and 5 denier polyester; binder fraction much less than 50%.

4. Batt made similarly to that of 3., above, except that fiber blending within the tow was improved.

5. Batt made primarily of 1.5 denier continuous filament polyester with top and bottom layers of continuous filament Type 254 binder.

6. Batt made similarly to that of 5., above, except webs of layered Type 254 binder in staple form (rather than continuous filament) were laid on the top and bottom.

7. Batt made of alternating layers of modified Type 254 binder, continuous filament (20/80 sheath/core, rather than 50/50) and 1.5 denier polyester, continuous filament.

8. Batt made form a single tow bundle that was a mixture of modified Type 254 binder (as in 7., above) and 1.5 denier polyester; binder fraction less than 25%.

The batts made to evaluate each of the eight variants were relatively small and had been painstakingly made using various combinations of production equipment, laboratory equipment and manual handling. All had deficiencies that, we believe, were representative of the concept; the samples had been skillfully made. All were characterized by very poor loft and an unacceptable, stiff hand. Hoechst Celanese and AI staff members agreed that a diligent effort had been made to pursue the binder-fiber/continuous-filament-batt concept, that the results were not encouraging and that any further work would be dependent upon new ideas and a larger development budget.

Realizing that bicomponent binder fiber technology could not be matched to the needs of the program, Hoechst Celanese explored the prospects for utilizing a sprayable binder resin that would not negatively affect: (1) FR, (2) water repellency, or (3) the hand of the batt. Rohm and Haas, manufacturers of the resin that Hoechst Celanese uses on commercial, continuous filament insulators, attempted to identify an acceptable resin formulation, but none was found. Hoechst Celanese, after several months of dialogue with Rohm and Haas, decided to abandon the spray resin approach in favor of a somewhat similar, but different, approach.

The third bonding method considered consisted of melt blowing an extremely lightweight, almost imperceptible web of polyester onto the top and bottom surfaces of the continuous filament batt. Hoechst Celanese enlisted the aid of two Charlotte (North Carolina) area firms in developing and evaluating this approach in the laboratory. Their initial results were encouraging; continuous filament batts made of 5 denier polyester, bonded using the melt blowing technique, were of very good overall quality. The bonded structure appeared to have more than adequate integrity and washing tests confirmed that this was so. Loft and hand were also surprisingly good. Although the amount of material (polyester) added to the batt was not measured, it was minimal, probably less than 5% of the batt weight. This led us to assume that the treatment would not significantly affect the flame resistance or the wetting resistance of the batt.

All who were involved were encouraged by the laboratory success of the melt blowing approach and plans were made to move the process into a production-scale tow spreading facility in the Charlotte area. Hoechst Celanese prepared a large quantity of 1.2 denier/filament, FR, polyester tow for the plant trial and, because the water repellent finish issue had not been resolved, conducted controlled finishing experiments during tow production. Installing the melt blowing apparatus at the cross-lapper output point on the tow spreading line had been acknowledged to be a logistical and engineering challenge. However, a few days before the scheduled trial, as preparation work was underway, the operator of the spreading line reevaluated the cost of his involvement and unilaterally canceled the trial. The only other tow spreading operator that would consider having the melt blowing apparatus installed on their line was Reliance Products Co. in Oakland, California. In spite of the complication that distance (Charlotte to Oakland) would add to the logistics, installing the equipment at Reliance was given serious consideration. However, the cost estimate made for the endeavor showed, without question, that it was not feasible and pursuit of the melt blowing approach had to be terminated.

The fourth and final method of batt stabilization tried was needling. It offered the distinct advantage of not adding material to the batt; most materials that might otherwise be added would negatively affect flame resistance and water repellency. However, an equally distinct disadvantage was foreseen; needling is a consolidation process and usually yields product of greater density than the 0.3 to 0.6 lb/ft^3 target range established for the program. Working with 3 denier/filament tow in the laboratory, Hoechst Celanese made a set of needled batt samples that indicated some promise for the approach. Subsequent evaluation in our laboratory showed that a reasonable compromise between machine-direction strength and density had been obtained. Strength was adequate and density determinations ranged from 0.57 to 0.80 lb/ft^3. The most satisfactory of the samples had an average density of 0.63 lb/ft^3.

E. Pilot Line Trial

Review of the experience and results of the laboratory needling trial led to the assessment that a needled continuous filament insulator would not fully satisfy all program targets, but would, nonetheless, constitute a credible solution. Consequently, an opening, spreading and needling trial was scheduled at Reliance Products Co. in Oakland and tow originally prepared for work with the melt blowing technique was shipped to them.

The results of the trial at Reliance Products were disappointing. None of 1.2 denier/filament, FR tow opened well, regardless of finish (several finish options were evaluated). Non-FR, 1.2 denier/filament tow,

with "standard silicone finish," was tried and it did perform well in the fiber opening section but none of the Hoechst Celanese staff members involved could explain the essential difference between the FR and the non-FR tow. Incomplete fiber opening resulted in a non-uniform web with poor loft and it could not, of course, be improved in subsequent processing steps. Nonetheless, the cross-lapped batt was needled in an attempt to learn as much as possible about needling continuous filament, small-fiber-diameter batt.

A representative sample of the insulating batt produced in the Oakland trial was evaluated in our laboratory. Our intention was to first measure performance properties for which we suspected shortfalls, and, since several shortfalls were found, not all properties originally of interest were measured. A summary of the properties measured is shown in Table 20, together with corresponding program target values. Comparison of density, compressional recovery, absorptive capacity and vertical flammability test results with target values confirms that the sample was not a worthwhile insulator candidate.

F. Conclusion of Continuous Filament Insulator Development

Extended correspondence between Hoechst Celanese and AI Research ensued following the opening, spreading and needling trial in Oakland. It had become apparent that not all properties targets could be met and a consensus evolved concerning a worthwhile compromise. Hoechst Celanese' inability to attain the desired level of water repellency with silicone finishes and silicone's apparent negative affect on flammability led to an approach that omitted water repellency from the list of objectives and sought to satisfactorily address all other objectives. Our combined judgement was that an FR polyester, continuous filament insulator that met all program targets except water repellency would fulfill otherwise unmet needs and be cost-effective; water repellency could be addressed in end-item design through appropriate selection of cover fabrics and/or membrane layers. Working to this premise, Hoechst Celanese staff members identified two phosphate-based, lubricating finishes that would facilitate tow opening and spreading. Each of these finishes appeared, as the result of laboratory work, to offer promise in terms of our revised purpose. However, in planning the next step, application of the finishes on a production-scale fiber spinning line, an unyielding obstacle was encountered. Hoechst Celanese' production commitments precluded spinning line interruptions for further development work for six-to-nine months.

Throughout the FR, continuous filament development work, unexpected, often unworkable situations and efforts to circumvent them were inevitably accompanied by delays and unanticipated costs. Thus, when Hoechst Celanese advised of a further six-to-nine month delay, at a point when the promise of technical success was clearly diminished, AI Research and Natick Center re-assessed the continuous

Table 20. ***Summary of Selected Properties; Experimental, Continuous Filament, Flame Resistant Thermal Insulation***

Performance Property	Program Target	Experimental, Continuous Filament Insulator
Density (lb/ft^3) (kg/m^3)	0.3 to 0.6 4.8 to 9.6	0.87 13.9
Compressional recovery (%)	$\geq$ 90	83
Absorptive capacity after 20 minutes of immersion	$\leq$150	699
Flammability per Federal Test Method 5903, in machine and cross-machine directions, respectively After flame (sec) After glow (sec) Char/destroyed length (inch) (cm)	 $\leq$ 2 $\leq$ 25 $\leq$ 5.5 $\leq$14.0	 28; 22 0; 0 7.2; 5.8 18.3; 14.7

Values shown are averages. Fewer test replications than usual were made because of obvious performance shortfalls. The minimum number of test replications was three.

filament portion of the program. It was agreed that the concept of an FR, continuous filament insulator had been thoroughly investigated, that the prospects for success in terms of original program goals were minimal and that a practical conclusion point had, in fact, been reached.

The development experience reported in this section will provide the basis for an appraisal of the long-term viability of the FR, continuous filament insulator concept, to be made in the concluding section (VIII).

5. PILOT LINE PRODUCTION OF THE STAPLE INSULATOR PROTOTYPE

Albany International's Primaloft production line in Albany, New York was used for a manufacturing trial of the blended-staple insulator prototype described at the conclusion of Section III. The Primaloft line was installed in 1988 to manufacture blended, thermally-bonded microfiber insulation and, as experience has been gained, has been improved through equipment modifications and additions. The general configuration of the line is not unique, but several component stations make it uniquely suited to the manufacture of blended, microfibrous batts. Fiber weighing, mixing, pre-opening and carding stations are all configured to efficiently process microfiber blends. An in-line, infrared/hot air oven is routinely used to effect bonding in blends containing bicomponent binder fiber.

Two AI Research Co. staff members and several members of the Primaloft management and manufacturing staff participated in an FR insulator prototype trial in June of 1993. Prior to the scheduled date, the efficacy of producer-applied water repellent finish on the P84 microfiber and macrofiber obtained for the prototype run had been confirmed through testing in our Mansfield, Massachusetts laboratory. The fiber blend used in the trial was exactly that described on the final page of Section III (page 39). Processing progressed with little difficulty and, after an initial period of adjustments to feed rate and oven temperature, insulating batt of excellent quality and of the desired areal density and loft was being produced. Several rolls, each 60 inches wide by approximately 20 yards long, were made and weight and loft were monitored using batt samples cut at each roll change. This sampling procedure precluded discontinuities within each roll. The samples taken between rolls yielded the following dimensional and weight characterization of the prototype material:

Areal density	3.9 to 4.2 oz/yd^2
Thickness	0.97 to 1.07 inches
Volume density	0.31 to 0.36 lb/ft^3

The program target for areal density was 4.0 oz/yd^2 and the volume density target was 0.3 to 0.6 lb/ft^3. Loft (thickness) was intentionally maximized, yielding volume densities at the lower end of the target range, for two reasons. The first was to provide an insulator with the greatest possible "insulating value/weight" efficiency and the second was to compensate for a small amount of compression-set anticipated as a result of roll-up and storage.

Several processing parameters that will be important in duplicating the pilot trial results were measured (and/or calculated) as follows:

Output rate of card	30 lb/hr
Output rate of cross-lapper	72 linear yd/hr
Oven air temperature	375°F
Dwell time in oven	4.5 minutes

The time/temperature combination shown above is more extreme than that which the Primaloft staff routinely uses to bond all-polyester batts with the same binder fiber component (Hoechst Celanese Type K54). In bonding all-polyester batts, the time/temperature combination must be carefully limited to effect bonding without unduly softening the primary polyester component and inducing loft loss. However, it was realized that the temperature resistance of P84 (it withstands continuous use at 550°F) obviated the loft loss consideration and so a greater amount of heat was applied in the interest of improved bonding. Handling the FR insulator after it cooled, just prior to roll-up, provided assurance that near-optimal bonding had been obtained. Subsequent testing, reported in a following section, confirmed the quality of the bonding.

A 0.4 oz/yd^2 nonwoven polyester scrim was continuously fed onto the cross-lapper, below the batt being formed, to ensure safe transit of the FR insulator through the remainder of the line to roll-up. The stabilization provided to the batt, especially at roll-up, was essential in obtaining a first quality, lofty prototype. This one-side scrim can be very easily removed from the FR insulator as it is unrolled, or perhaps preferably, after it has been unrolled and cut into test specimens or pieces for end-item fabrication. <u>It is important to note that the scrim is not flame-resistant or water resistant and is intended to be removed for testing and for use</u>.

On October 25, 1993, four of the 60-inch wide rolls of 4 oz/yd^2, staple-based, FR insulator prototype were delivered to Natick. Each roll was approximately 20 yd long, yielding about 33 yd^2 per roll and about 130 yd^2 total. Prior to this shipment, additional prototype material taken from rolls virtually identical to those shipped was subjected to a wide range of physical properties tests. This testing, which will be reported in a section that follows, confirmed that insulator properties met or exceeded all program target values.

6. LABORATORY CHARACTERIZATION OF PILOT LINE SAMPLES OF THE STAPLE INSULATOR PROTOTYPE

A. Performance Goals and Test Methods

Various laboratory test methods especially suitable for high loft insulators were adopted during the course of two previous Natick studies performed at Al Research Co.[1,2,3] These studies have been a source of important reference data for both Natick and Al Research. Most test methods that were specified in the Work Statement of the subject contract (except those relating to flame resistance) were among those adopted during the earlier work. Thus, their relevance had been proven and the data generated using them would be directly comparable to existing reference data. The Work Statement included the following specification of test methods and properties targets:

"1. Thermal Conductivity: ≤ 0.300 Btu-in/ft^{2}hr-°F (0.043 W/m-°C) @ 0.5 lb/ft^3 (8.0 Kg/m^3) as measured by ASTM C518.

2. Density: 0.3 to 0.6 lb/ft^3 (4.8- 9.6 Kg/m^3), thickness measured at 0.002 psi (0.0138 kPa).

3. Launderability: 3 cycles with ≤ 25% thickness decrease, ≤ 10% thermal resistance decrease, and shrinkage ≤ 5%. Prepared and laundered as specified in Mil-B-41826G; Batting, Synthetic Fibers, Polyester, (Unquilted and Quilted), Sections 4.5.3 and 4.5.4 using the Cotton Procedure Method 5556 of FED-STD-191. The batting appearance must meet the Photographic Rating Standard for Fiberfill Durability according to ASTM D4770 as specified in MIL-B-41826G, Section 4.5.5. See Appendix II. Thickness measured at 0.002 psi (0.0138 kPa), thermal resistance measured by ASTM C518.

4. Work to Compress: ≤ 2.75 lb/in (0.113 N-m), per Mil-B-41826G, Section 4.5.2 except stress limits of 0.002 psi (0.0138 kPa) and 5.0 psi (34.5 kPa).

5. Resilience: ≥55 %; work of recovery divided by work to compress. Work of recovery per MIL-B-41826G, Section 4.5.2 except stress limits of 0.002 psi (0.0138 kPa) and 5.0 psi (34.5 kPa).

6. Compressional Recovery: ≥ 90% from a stress level of 5.0 psi (34.5 kPa) per MIL-B-41826G, Section 4.5.2.

7. Absorptive Capacity: not more than 150% water retention after 20 minutes based on ASTM D1117 as modified in Natick Technical Report TR-86/021L p. 88.

8. Compressional Strain: ≥ 95% @ 5.0 psi (34.5 kPa) per MIL-B-41826G, Section 4.5.2 except initial thickness at 0.002 psi (0.0138 kPa).

9. Wet loft retention: 95% with 20 min. wetting and ≥ 50% over 6 hrs. based on ASTM D1117 as modified in Natick TR086/021L p. 88.

10. Flammability: A variation of Fed. Test Method 5907 using a 90 second low energy methenamine pill and Fed. Test Method 5903. The samples should meet the following requirements before and after washing: After flame: 0; After glow: no more than 25 seconds; Char length: no more than 3.5 inches. In addition there should be no flame propagation and no melting or dripping of the sample."

The above test methods were followed explicitly to obtain the data that follows. However, in a few instances, minor deviations in procedure were necessary; these will be explained below. In other instances, information beyond that given in the test method specified may be helpful; such information will also be reported below. A change in flammability target values, for the continuous filament insulator candidate only, was made after work had begun (this was reported in Section II). The original flammability targets, as shown in 10., above; apply to the staple insulator candidate that is the subject of this section.

Thermal conductivity and thermal resistance measurements were the only ones not made in our Mansfield laboratory; they were made by Holometrix, Inc. (formerly Dynatech) of Bedford, Massachusetts. Two instrument types were used: (1) the Holometrix Rapid K, which has a 12 x 12 inch sample area with a 4 x 4 inch measuring area in the center and (2) the Holometrix R-matic, which accommodates a 24 x 24 inch sample and has a 12 x 12 inch measuring area in the center. Both instruments are plate-to-plate types that are used in compliance with ASTM C518. The smaller Rapid K apparatus was used to evaluate most insulator test specimens, with heat flow downward, throughout the developmental portion of the work and again at the conclusion. However, the larger R-matic apparatus was found to be more suitable for measurement of the 24 x 24 inch quilted laundering specimens and so was used to determine change in thermal resistance due to laundering. The R-matic can be operated only in the heat flow upward mode. In all thermal conductivity/resistance tests, with both instrument types, the temperature of the hotter plate was 100°F and that of the cooler was 50°F.

The laundering method used as part of the test procedure for characterizing insulator launderability, or resistance to laundering, differed slightly in detail from that specified in the Work Statement. However, the six-step wash/rinse method given as the "Cotton Procedure" of Method 5556 of Federal Test Method Standard No. 191A was, for all practical purposes, duplicated. A Powercom wash wheel, Model No. 24-20A with programmable cycling, was used. Differences in detail from the sample preparation (per MIL-B-41826G) and laundering method specified were as follows:

1. The cover fabric used to prepare the quilted laundering samples was a 2.0 oz/yd^2 nylon taffeta described in MIL-C-21852, Type III, Class 1. We were advised that use of this cover fabric is the current practice at Natick Center.

2. The detergent used was Igepon 73, 10 g in the first wash step and 6 g in the second step. Sodium silica fluoride sour, 24 g, was used in the fifth (rinse) step. These changes were also made to comply with Natick Center's current practice.

3. Following laundering, water was extracted from the load using a commercial heavy duty machine set on the spin cycle. The wash load was divided into two equal portions for this step and each was spun for 3 minutes. This approximated the effect of a commercial extractor, which we did not have available.

All other test methods cited in the Work Statement were employed and followed exactly as specified.

B. Laboratory Evaluation; Discussion of Results

The FR staple insulator development effort described in preceding sections enabled us to select fiber and blend ratio for the prototype insulator with confidence that program performance targets would, to a great extent, be satisfied. The final step in the process was to be laboratory evaluation of the prototype, using the test methods described above, to verify that desired performance characteristics had been attained. The results of the prototype evaluation follow in a set of data compilations that include: individual test results, averaged results and program target values. Although most of these results can be readily interpreted and compared directly to program targets, brief commentary will, in several cases, be helpful.

The compressional and compressional recovery characteristics of the staple FR insulator are given in companion Tables 21 and 22. All values shown meet or exceed program targets and, taken together, describe a material that is soft to the touch and yet has very good compressional recovery and resilience. The hand of the material is down-like.

Water absorptive capacity and wet loft retention values, reported in Tables 23 and 24, respectively, show that the insulator prototype meets or exceeds the program's difficult wetting resistance objectives.

Three sets of vertical flammability test results (per Method 5903.1 of Fed. Std. No. 191A) obtained for the prototype insulator are shown in Table 25. Prototype samples were tested before laundering, after laundering and again after laundering and additional rinsing. The results obtained prior to laundering agree well with those reported in a previous section for small hand-samples made of the prototype blend during the development phase. Afterflame times, afterglow times and char lengths are well within the target limits. However, the "after laundering" data set in Table 25 includes values for individual specimens that greatly exceed afterflame and char length objectives. These results were, of course, unexpected and led to immediate, careful inspection of the remaining laundered material. Some retention of fatty-acid based detergent in the insulator was among the causes suspected first and hand rinsing of a small laundered sample-piece confirmed the presence of detergent. Another set of laundered vertical flammability specimens was then thoroughly rinsed, by hand, in water. This additional rinsing, unlike the first rinsing that was part of the "Cotton Procedure" of Method 5556, was done without cover fabric over the insulator. Although further rinsing obviously removed detergent (the rinse water became somewhat filmy and bubbly), we could not be sure that all detergent was eliminated. The samples had been dried after the apparently incomplete "Cotton Procedure" rinse and repeated additional rinsing of the open, unquilted insulator samples tended to distort them and, it seemed, remove only trace amounts of detergent. Consequently, rinsing was stopped at a point judged to be that of diminishing returns and the samples were dried and subjected to vertical flammability testing. The results are given in Table 25 under the heading: "After Laundering, Additional Rinsing." Six specimens were tested in each principal fabric direction and nine of the twelve afterflame/afterglow/char-length data sets are well within target values and close to or, in some cases, the same as "before laundering" data. Three of the twelve data sets include long afterflame times and extreme char lengths that are, almost certainly, the result of residual detergent in the insulator.

Table 21 E. *Compression Strain and Recovery of Flame Resistant, Staple-Fiber Insulation*[a]

Sample No.	Initial Thickness (inch)	Areal Density (oz/yd^2)	Volume Density (lb/ft^3)	Compressional Strain at 5 lb/in^2 (%)	Compressional Recovery (%)
1	0.76	3.94	0.42	97	94
2	0.84	3.53	0.35	97	100
3	0.76	3.91	0.40	97	93
4	0.76	3.71	0.41	97	100
5	0.78	3.73	0.37	97	92
Average	0.78	3.76	0.39	97	96
Program Target	--	4, nominal	0.3 to 0.6	≥95	≥90

a. Tests were performed per MIL-B-41826G, Section 4.5.2, except stress limits of 0.002 and 5 lb/in^2 were employed.

Table 22 E. *Work to Compress, Work to Recover, and Resilience of Flame Resistant, Staple-Fiber Insulation*[a]

Sample No.	Initial Thickness (inch)	Areal Density (oz/yd^2)	Volume Density (lb/ft^3)	Work to Compress to 5 lb/in^2, W_c (lb-in)	Work to Recover to Zero Stress, W_r (lb-in)	Resilience, $W_r/W_c \times 100$ (%)
1	0.80	3.63	0.38	1.37	1.14	83
2	0.96	4.27	0.37	1.32	1.09	83
3	0.86	3.95	0.38	1.29	0.99	77
4	0.80	3.67	0.38	1.33	1.07	80
5	0.86	3.67	0.36	1.38	1.03	75
Average	0.86	3.84	0.37	1.34	1.06	80
Program Target	--	4, nominal	0.3 to 0.6	≤2.75	--	≥55

a. Tests were performed per MIL-B-41826G, Section 4.5.2, except stress limits of 0.002 and 5 lb/in^2 were employed.

Table 21 SI. ***Compression Strain and Recovery of Flame Resistant, Staple-Fiber Insulation[a]***

Sample No.	Initial Thickness (cm)	Areal Density (g/m^2)	Volume Density (kg/m^3)	Compressional Strain at 34 kPa (%)	Compressional Recovery (%)
1	1.93	134	6.7	97	94
2	2.13	120	5.6	97	100
3	1.93	132	6.4	97	93
4	1.93	126	6.6	97	100
5	1.98	126	5.9	97	92
Average	1.98	128	6.2	97	96
Program Target	--	136, nominal	4.8 to 9.6	≥95	≥90

a. Tests were performed per MIL-B-41826G, Section 4.5.2, except stress limits of 0.014 and 34 kPa were employed.

Table 22 SI. ***Work to Compress, Work to Recover, and Resilience of Flame Resistant, Staple-Fiber Insulation[a]***

Sample No.	Initial Thickness (cm)	Areal Density (g/m^2)	Volume Density (kg/m^3)	Work to Compress to 34 kPa, W_c (N-m)	Work to Recover to Zero Stress, W_r (N-m)	Resilience, $W_r/W_c \times 100$ (%)
1	2.03	123	6.1	0.155	0.129	83
2	2.44	145	5.9	0.149	0.123	83
3	2.18	134	6.1	0.146	0.112	77
4	2.03	124	6.1	0.150	0.121	80
5	2.18	124	5.8	0.156	0.116	75
Average	2.18	130	5.9	0.151	0.120	80
Program Target	--	136, nominal	4.8 to 9.6	≤0.311	--	≥55

a. Tests were performed per MIL-B-41826G, Section 4.5.2, except stress limits of 0.014 and 34 kPa were employed.

Table 23. *Water Absorptive Capacity[a] of Flame Resistant, Staple-Fiber Insulation After 20 Minutes of Immersion*

Sample No.	Wet Weight (g)	Absorptive Capacity (%)
1	1.94	129
2	1.65	110
3	1.84	123
4	2.02	135
5	1.93	129
6	1.70	113
7	1.95	130
8	2.05	137
9	1.75	117
10	1.73	115
Average	1.86	124
Program Target	--	≤150

a. Test was performed per ASTM Method D1117 modified in Natick Technical Report TR-86/021L, p. 88. Absorptive capacity was obtained by dividing wet weight by dry weight and multiplying by 100 to obtain value as a percentage. All of the above is based upon a dry weight of 1.50 g.

Table 24 E. Wet Loft Retention[a] of Flame Resistant, Staple-Fiber Insulation

Sample No.	Dry Thickness (inches)	Dry Density (lb/ft^3)	After 20 Minutes Immersion				After 6 Hours Immersion			
			Thickness (inches)	Loft Retention (%)	Density (lb/ft^3)	Density Increase (%)	Thickness (inches)	Loft Retention (%)	Density (lb/ft^3)	Density Increase (%)
1	0.84	0.35	0.74	88	0.50	43	0.72	85	0.65	86
2	0.80	0.35	0.78	97	0.40	14	0.66	83	0.65	86
3	0.78	0.39	0.74	95	0.56	44	0.68	87	0.70	79
4	0.78	0.39	0.76	97	0.50	28	0.70	89	0.67	72
5	0.78	0.43	0.76	97	0.50	16	0.66	85	0.72	67
Average	0.80	0.38	0.76	95	0.49	29	0.68	86	0.68	79
Program Target	--	0.3 to 0.6	--	≥95	--	--	--	≥50	--	--

a. Test was performed per ASTM Method D1117 as modified in Natick Technical Report TR086/021L, p.88. The areal density of the test samples was approximately 4 oz/yd^2.

Table 24 SI. Wet Loft Retention[a] of Flame Resistant, Staple-Fiber Insulation

Sample No.	Dry Thickness (cm)	Dry Density (kg/m^3)	After 20 Minutes Immersion				After 6 Hours Immersion			
			Thickness (cm)	Loft Retention (%)	Density (kg/m^3)	Density Increase (%)	Thickness (cm)	Loft Retention (%)	Density (kg/m^3)	Density Increase (%)
1	2.13	5.6	1.88	88	8.0	43	1.83	85	10.4	86
2	2.03	5.6	1.98	97	6.4	14	1.68	83	10.4	86
3	1.98	6.2	1.88	95	9.0	44	1.73	87	11.2	79
4	1.98	6.2	1.93	97	8.0	28	1.78	89	10.7	72
5	1.98	6.9	1.93	97	8.0	16	1.68	85	11.5	67
Average	2.03	6.1	1.93	95	7.8	29	1.73	86	10.9	79
Program Target	--	4.8 to 9.6	--	≥95	--	--	--	≥50	--	--

a. Test was performed per ASTM Method D1117 as modified in Natick Technical Report TR086/021L, p.88. The areal density of the test samples was approximately 136 g/m^2.

Table 25. *Vertical Flammability Test Results for Flame Resistant, Staple-Fiber Insulation*[a]

	Before Laundering				After Laundering[c]				After Laundering,[d] Additional Rinsing			
	Afterflame	Afterglow	Char[b] Length		Afterflame	Afterglow	Char Length		Afterflame	Afterglow	Char Length	
Test Direction	(sec)	(sec)	(inch)	(cm)	(sec)	(sec)	(inch)	(cm)	(sec)	(sec)	(inch)	(cm)
Machine	0	3	1.5	3.8	29	0	10.0	25.4	0	3	2.0	5.1
	0	2	1.2	3.0	0	4	2.5	6.4	0	2	1.0	2.5
	0	4	1.8	4.6	0	3	2.2	5.6	0	4	1.2	3.0
	0	4	1.5	3.8	8	0	3.5	8.9	0	4	2.2	5.6
	0	3	1.5	3.8	0	5	2.5	6.4	0	2	1.8	4.6
									31	0	8.2	20.8
Average	0	3	1.5	3.8	7	2	4.1	10.4	5	2	2.6	6.6
Cross-machine	0	3	1.0	2.5	26	0	8.0	20.3	0	2	1.0	2.5
	0	3	1.0	2.5	23	0	7.5	19.0	13	0	7.2	18.3
	0	2	0.8	2.0	29	0	11.0	27.9	21	0	7.5	19.0
	0	2	1.0	2.5					0	6	2.0	5.1
	0	2	0.8	2.0					0	5	1.8	4.6
									0	5	2.2	5.6
Average	0	2	0.9	2.3	26	0	8.8	22.4	6	3	3.6	9.1
Program Target	0	≤25	≤3.5	≤8.9	0	≤25	≤3.5	≤ 8.9	0	≤25	≤3.5	≤8.9

a. Per Method 5903.1, "Flame Resistance of Cloth, Vertical," of Fed. Std. No. 191A.
b. Shrinkage typically comprised about 0.5 inch (1.3 cm) of these char lengths.
c. Inability to remove all soap in rinsing judged to negatively affect results.
d. Measurements after additional water rinsing on batt samples that had dried and were then removed from quilted cover fabric. Although these results are mixed, data for some samples agrees well with "before laundering" data, supporting judgement that incomplete soap removal was responsible for the decrease in flame resistance after laundering.

[1]No melting or dripping was observed in any of the tests reported above. In all "Before Laundering" tests and in most "After Laundering" tests, no flame propagation was observed. However, some of the "After Laundering" samples, those which exhibited extreme afterflame times due to incomplete rinsing, did propagate flame.

Flammability test results obtained using two variations of the methenamine ignition-pill test (of Method 5907 of Fed. St. No. 191A) are reported in Table 26. Data is shown for two sample sets, one tested without having been laundered and the other tested after laundering. The laundered samples were subjected to additional rinsing, beyond that of the "Cotton Procedure," as described above. All data points in Table 26, under both "Before Laundering" and "After Laundering" headings, are well within target values. The lack of any negative influence due to detergent retention, which affected three of twelve well-rinsed vertical flammability specimens, may be attributed to two factors: (1) the lesser severity of the pill tests in comparison to the vertical flammability test for lightweight batts of this type (based upon our observations throughout the program) and/or (2) the secondary rinsing, in this case, may have been effective for all samples.

Dimensional and appearance changes in the prototype insulator due to laundering are reported in Table 27. Based upon thickness decrease, planar shrinkage and appearance rating, the prototype's resistance to degradation during laundering is very good. However, the three planar shrinkage values shown range from 7.4 to 7.8% and the program target is $\leq$ 5%. The apparent shortfall warrants comment. Planar shrinkage measurements were made, per MIL-B-41826G, on fabric-covered, quilted insulation samples and the values obtained are the net result of small amounts of shrinkage in insulation, cover fabric, and sewing thread. Initial assembly tightness and sewing thread tension also appeared to affect the results. Another factor, obvious at the time of measurement, was that the 7.6% average shrinkage value could be reduced to about the 5% target level by smoothing small gathers in the laundered sample. Awareness of these variables and of the obviously good, overall laundering durability exhibited by the insulator prototype made the significance of the apparent shrinkage shortfall seem minor.

Thermal conductivity and thermal resistance of the staple-fiber insulator, measured before and after laundering, are given in Table 28. The data compares favorably with program targets (shown in the table) for the insulating performance of new and laundered samples. However, the data set contains a minor flaw that must be mentioned. A very small <u>increase</u> in insulating performance following laundering is indicated, although we believe that a more accurate result would indicate a similarly small decrease in insulating performance (one that would be within the target limit). Through laboratory error, the plate gap of the thermal conductivity apparatus used to obtain the "after laundering" data was set to the "before laundering" sample thickness. The error was not apparent because complete sample-to-plate contact was made using this gap setting. The average "before laundering" thickness was 0.78 inch and the average "after laundering" thickness was 0.71 inch; the 0.07 inch difference could not be detected without the standard measuring pressure of 0.002 lb/in^2 being applied to the "after laundering" sample. The very fact

Table 26. *Flammability Test Results for Flame-Resistant, Staple-Fiber Insulation Obtained Using Methenamine Ignition Pill*[a]

Test Direction	Before Laundering				After Laundering[b]			
	After-flame (sec)	After-glow (sec)	Char Length (inch)	Char Length (cm)	After-flame (sec)	After-glow (sec)	Char Length (inch)	Char Length (cm)
Machine; sample folded at 45°; ignition pill at fold	0	0	0.6	1.5				
	0	0	0.7	1.8				
	0	0	1.2	3.0				
	0	0	0.6	1.5				
	0	0	0.9	2.3				
Average	0	0	0.8	2.0	–	–	–	–
Cross-machine; sample folded at 45°; ignition pill at fold	0	0	0.7	1.8	0	0	0.8	2.0
	0	0	1.2	3.0	0	0	0.8	2.0
	0	0	1.1	2.8	0	0	0.9	2.3
	0	0	1.3	3.3	0	0	0.9	2.3
	0	0	1.8	4.6	0	0	0.9	2.3
					0	0	1.0	2.5
					0	0	0.6	1.5
					0	0	0.5	1.3
					0	0	0.5	1.3
Average	0	0	1.2	3.0	0	0	0.8	2.0
Horizontal sample; ignition pill at center	0	0	1.2	3.0	0	0	1.0	2.5
	0	0	1.0	2.5	0	0	1.4	3.6
	0	0	1.1	2.8	0	0	2.4	6.1
	0	0	1.0	2.5	0	0	1.0	2.5
	0	0	1.2	3.0	0	0	1.5	3.8
					0	0	1.8	4.6
					0	0	1.5	3.8
					0	0	1.0	2.5
					0	0	1.0	2.5
Average				2.8	0	0	1.4	3.6
Program Target	0	≤25	≤3.5	≤8.9	0	≤25	≤3.5	≤8.9

a. Per Method 5907, "Flammability Test for Sleeping Bag Cloths; Tablet Method," of Fed. Std. No. 191A, as modified in the Work Statement of the subject contract. The Work Statement specified that: "No test cloth will be used over the batting sample." It also added the third test format, that of the horizontal sample with the ignition pill placed in the center.

b. Inability to remove all soap in rinsing apparently affected the vertical flammability test results (Table 25) and so samples for the tests reported above received two extra rinses after removal from the quilted cover fabric. Laundered batt was not available in enough quantity to permit 45° fold testing in both principal batt directions.

Table 27. *Changes in Flame-Resistant, Staple-Fiber Insulation, Sewn in Cover Fabric, Due to Laundering*[a]

Sample No.	Initial[b] Thickness		Thickness[b] After Wash		Thickness[b] Decrease (%)	Average[c] Planar Shrinkage (%)	Appearance[d] Rating After Wash
	(inch)	(cm)	(inch)	(cm)			
1	0.75	1.90	0.72	1.83	4.0	7.4	4
2	0.76	1.93	0.71	1.80	6.6	7.6	4
3	0.83	2.11	0.71	1.80	14.4	7.8	4
Average	0.78	1.98	0.71	1.80	9.0	7.6	4
Program Target	--	--	--	--	≤25	≤5	≥4

a. Quilted samples prepared, laundered and measured in accord with MIL-B-41826G, using the Cotton Procedure of Method 5556 of Fed. Std. 191; 3 laundering/drying cycles.
b. Average of thicknesses measured at four locations on each sample.
c. Average of three machine-direction and three cross-direction measurements per sample.
d. Appearance judged using the Photographic Rating Standard of ASTM D4770 as specified in MIL-B-41826G.

Table 28 E. *Thermal Conductivity and Thermal Resistance of Flame-Resistant, Staple-Fiber Insulation; Individual Test Results*

Sample Description	Test Format	Test No.	Test Thickness (inch)	Test Density (lb/ft^3)	Apparent[b] Thermal Conductivity ($Btu\text{-}in/hr\text{-}ft^2\text{-}^\circ F$)	Thermal[c] Resistance ($hr\text{-}ft^2\text{-}^\circ F/Btu$)
Batt plied to obtain 2 inch thickness and 0.5 lb/ft^3 density (standard values)	12 x 12 inch; heat flow down	1	1.94	0.50	0.293	6.61
		2	1.93	0.50	0.296	6.51
		Average	1.94	0.50	0.294	6.56
Laundering samples; 4 oz/yd^2 batt sewn between cover fabric; <u>before laundering</u>	24 x 24 inch; heat flow up	1	0.77	0.99	0.296	2.60
		2	0.76	0.99	0.293	2.60
		3	0.83	0.95	0.299	2.78
		Average	0.79	0.98	0.296	2.66
Laundering samples described above; after 3 laundering cycles per Method 5556[a]	24 x 24 inch; heat flow up	1	0.77	1.01	0.291	2.65
		2	0.76	1.00	0.290	2.62
		3	0.83	0.95	0.293	2.83
		Average	0.79	0.99	0.291	2.70

a. Changes in laundering-sample dimensions are reported in Table 27. The changes include an average thickness change of - 9.0%, obtained using a measuring platen pressure of 0.002 lb/in^2. This thickness change was not apparent as the samples were installed in the thermal conductivity test apparatus and so the "after laundering" test thicknesses are, when reported to two decimal places, unchanged from the "before laundering" test thicknesses.

b. The program target is: $\leq$0.300 $Btu\text{-}in/hr\text{-}ft^2\text{-}^\circ F$.

c. The program target for thermal resistance after laundering is a decrease from the "before laundering" value $\leq$10%.

Table 28 SI. ***Thermal Conductivity and Thermal Resistance of Flame-Resistant, Staple-Fiber Insulation; Individual Test Results***

Sample Description	Test Format	Test No.	Test Thickness (cm)	Test Density (kg/m^3)	Apparent[b] Thermal Conductivity (W/m-K)	Thermal[c] Resistance ($K\text{-}m^2/W$)
Batt plied to obtain 5.1 cm thickness and 8.0 kg/m^3 (standard values)	30.5 x 30.5 cm; heat flow down	1	4.93	8.0	0.042	1.163
		2	4.90	8.0	0.043	1.146
		Average	4.92	8.0	0.042	1.154
Laundering samples; 136 g/m^2 batt sewn between cover fabric; <u>before laundering</u>	61 x 61 cm; heat flow up	1	1.96	15.9	0.043	0.458
		2	1.93	15.9	0.042	0.458
		3	2.11	15.2	0.043	0.489
		Average	2.00	15.7	0.043	0.468
Laundering samples described above; after 3 laundering cycles per Method 5556[a]	61 x 61 cm; heat flow up	1	1.96	16.2	0.042	0.466
		2	1.93	16.0	0.042	0.461
		3	2.11	15.2	0.042	0.498
		Average	2.00	15.8	0.042	0.475

a. Changes in laundering-sample dimensions are reported in Table 27. The changes include an average thickness change of - 9.0%, obtained using a measuring platen pressure of 0.014 kPa. This thickness change was not apparent as the samples were installed in the thermal conductivity test apparatus and so the "after laundering" test thicknesses are, when reported to two decimal places, unchanged from the "before laundering" test thicknesses.
b. The program target is: $\leq$0.043 W/m-K.
c. The program target for thermal resistance after laundering is a decrease from the "before laundering" value $\leq$10%.

that the laundered samples could be tested properly at their original thicknesses satisfied us that further evaluative efforts were not necessary.

The foregoing discussion of test results for the staple insulator prototype (reported in preceding Tables 21 through 28) has, for the sake of complete reporting, been directed toward various matters of detail, small deviations in test method and anomalous data points. In the section that follows, the laboratory data will be summarized and considered in terms of the performance potential it demonstrates.

7. SUMMARY OF LABORATORY PERFORMANCE AND ASSESSMENT OF OVERALL POTENTIAL OF THE STAPLE INSULATOR

A. Performance Summary and Performance Related to Cost

Laboratory characterization of the staple based, flame-resistant, insulator prototype is summarized in Table 29, which also includes the program target values given in the subject contract. These program targets, taken together, describe an insulator that has:

1. Insulating efficiency, on a weight basis, similar to that of waterfowl down,

2. Down-like compressional characteristics and hand,

3. Excellent resistance to wetting and to loss of loft when wet, outperforming down and almost all synthetic insulators in this regard,

4. A high degree of flame resistance, and

5. Durability, as determined by exposure to Military field laundering.

In addition to specific target values, a further, non-quantified requirement expressed in the contract was that the insulator "be obtainable at reasonable cost." Responding to the cost issue added complexity to several technical choices and required some minor performance compromise. However, as the summary of properties in Table 29 shows, virtually all specific performance targets have been met by the staple insulator prototype and, consequently, it does possess the unique, five-attribute combination outlined above.

The degree of success attained in addressing the "reasonable cost" requirement can be judged from review of Table 30, in which information for two currently used, flame-resistant insulators, a needled aramid and a needled novoloid batting, is compared with corresponding information for the prototype. Although neither reference material is directly comparable to the new, high loft, FR insulator, they are, to the best of our knowledge, the most relevant cost benchmarks available. Three "Insulator" entries for the prototype material are shown in the table; they differ only in degree of compression (and, consequently,

Table 29 E. ***Staple Based, Flame-Resistant, High Efficiency Thermal Insulation; Comparison of Performance Properties with Program Targets***

Performance Property	Program Target	Staple Based Insulator
Thermal conductivity (Btu-in/hr-ft^2-°F)	≤0.300	0.294
Density (lb/ft^3)	0.3 to 0.6	0.38
Launderability		
Thickness decrease (%)	≤25	9.0
Thermal resistance decrease (%)	≤10	1.5, increase
Planar shrinkage (%)	≤5	7.6
Appearance, per ASTM D4770 and MIL-B-41826G	Acceptable, per references	Acceptable, per references
Work to compress (lb-in)	≤2.75	1.34
Resilience (%)	≥55	80
Compressional strain (%)	≥95	97
Compressional recovery (%)	≥90	96
Absorptive capacity after 20 minutes of immersion (%)	≤150	124
Wet loft retention		
After 20 minutes immersion (%)	≥95	95
After 6 hours immersion (%)	≥50	86
Flammability per Federal Test Method 5903, in machine and cross-machine directions, respectively		
After flame (sec)	0	0;0
After glow (sec)	≤25	3;2
Char/destroyed length (inch)	≤3.5	1.5; 0.9
Flame propagation	None	None
Melting/dripping	None	None
Flammability per Federal Test Method 5907, specimen folded 45°, in machine and cross-machine directions, respectively		
After flame (sec)	0	0;0
After glow (sec)	≤25	0;0
Char/destroyed length (inch)	≤3.5	0.8; 1.2
Flammability per Federal Test Method 5907, horizontal specimen, source in center		
After flame (sec)	0	0
After glow (sec)	≤25	0
Char/destroyed length (inch)	≤3.5	1.1

Table 29 SI. ***Staple Based, Flame-Resistant, High Efficiency Thermal Insulation; Comparison of Performance Properties with Program Targets***

Performance Property	Program Target	Staple Based Insulator
Thermal conductivity (W/m-K)	≤0.043	0.042
Density (kg/m^3)	4.8 to 9.6	6.1
Launderability		
Thickness decrease (%)	≤25	9.0
Thermal resistance decrease (%)	≤10	1.5, increase
Planar shrinkage (%)	≤5	7.6
Appearance, per ASTM D4770 and MIL-B-41826G	Acceptable, per references	Acceptable, per references
Work to compress (N-m)	≤0.311	0.151
Resilience (%)	≥55	80
Compressional strain (%)	≥95	97
Compressional recovery (%)	≥90	96
Absorptive capacity after 20 minutes of immersion (%)	≤150	124
Wet loft retention		
After 20 minutes immersion (%)	≥95	95
After 6 hours immersion (%)	≥50	86
Flammability per Federal Test Method 5903, in machine and cross-machine directions, respectively		
After flame (sec)	0	0;0
After glow (sec)	≤25	3;2
Char/destroyed length (cm)	≤8.9	3.8; 2.3
Flame propagation	None	None
Melting/dripping	None	None
Flammability per Federal Test Method 5907, specimen folded 45°, in machine and cross-machine directions, respectively		
After flame (sec)	0	0;0
After glow (sec)	≤25	0;0
Char/destroyed length (cm)	≤8.9	2.0; 3.0
Flammability per Federal Test Method 5907, horizontal specimen, source in center		
After flame (sec)	0	0
After glow (sec)	≤25	0
Char/destroyed length (cm)	≤8.9	2.8

Table 30E. ***Materials Cost Related to Thermal Resistance for the Newly Developed, Staple-Fiber, FR Insulator and Two FR Insulators Now in Use***

Insulator	Areal Density (oz/yd^2)	Thickness (inch)	Volume Density (lb/ft^3)	Approximate[b] Thermal Resistance, R ($hr\text{-}ft^2\text{-}°F/Btu$)	Materials[c] Cost ($/lb)	Materials Cost per R ($\$/yd^2/R$)
Needled Nomex aramid batting per MIL-B-81813B	4.3[a]	0.26[a]	1.38	0.99	10.30	2.80
Needled Kynol novoloid batting per MIL-B-81813B	4.3[a]	0.26[a]	1.38	0.99	8.00	2.17
Newly developed, staple-based prototype with loft as measured	4.3	0.94	0.38	2.70	14.50[d]	1.44
Staple prototype, as above, slightly compressed	4.3	0.72	0.50	2.45	14.50[d]	1.59
Staple prototype, as above, greater compression	4.3	0.58	0.62	2.12	14.50[d]	1.84

a. Median value of the range specified in MIL-B-81813B.

b. All R values, except that of the new insulator candidate at 0.50 lb/ft^3 (2.45 R, measured), are conservative estimates based upon previously obtained data.

c. Based upon prices paid for fiber at the outset of the program in mid-1992.

d. Composite cost of the three-fiber blend determined from the following: P84 macrofiber (60%) @ $15.00/lb, P84 microfiber (22%) @ $24.00/lb and binder fiber (18%) @ $1.50/lb.

Table 30 SI. ***Materials Cost Related to Thermal Resistance for the Newly Developed, Staple-Fiber, FR Insulator and Two FR Insulators Now in Use***

Insulator	Areal Density (g/m^2)	Thickness (cm)	Volume Density (kg/m^3)	Approximate[b] Thermal Resistance ($K\text{-}m^2/W$)	Materials[c] Cost ($/kg)	Relative Materials Cost ($\$/m^2/K\text{-}m^2/W$)
Needled Nomex aramid batting per MIL-B-81813B	146[a]	0.66[a]	22.1	0.174	22.70	19.03
Needled Kynol novoloid batting per MIL-B-81813B	146[a]	0.66[a]	22.1	0.174	17.60	14.75
Newly developed, staple-based prototype with loft as measured	146	2.39	6.1	0.475	31.90[d]	9.78
Staple prototype, as above, slightly compressed	146	1.83	8.0	0.431	31.90[d]	10.80
Staple prototype, as above, greater compression	146	1.47	9.9	0.373	31.90[d]	12.50

a. Median value of the range specified in MIL-B-81813B.

b. All R values, except that of the new insulator candidate at 8.0 kg/m^3 (0.431 $K\text{-}m^2/W$, measured), are conservative estimates based upon previously obtained data.

c. Based upon prices paid for fiber at the outset of the program in mid-1992.

d. Composite cost of the three-fiber blend determined from the following: P84 macrofiber (60%) @ $33.04/kg, P84 microfiber (22%) @ $52.86/kg and binder fiber (18%) @ $3.30/kg.

volume density). If one prototype compression level is to be selected for comparison, that which corresponds to a density of 0.5 lb/ft^3 is perhaps most representative of in-use density and so makes the best choice. Although the new FR insulator has a greater materials cost than either reference material, its thermal resistance (on an equivalent weight basis) is approximately 2.5 times greater than that of both currently used materials. This cost/insulating-capacity balance is reported in terms of "Materials Cost per R" (Table 30) and the advantage is clearly in favor of the new FR insulator. Other, less easily quantifiable, performance comparisons among the prototype and the needled insulators of MIL-B-818138 are also important in any consideration of cost or value. The minimal weight, the down-like hand and the very high degree of wetting resistance exhibited by the new insulator add to its advantage over the needled FR materials and further increase its relative worth.

Another performance / cost consideration is the latitude that exists for changing the P84 microfiber / P84 macrofiber proportion from the 22/60 (microfiber / macrofiber) ratio of the prototype. As was reported in Section III, *Staple Insulator Development*, the flammability resistance of P84 blends was not sensitive to blend ratio, unlike virtually all other microfiber / macrofiber blends that were considered. This finding will allow the insulating performance / cost balance to be adjusted with a minimum of further development work. Increasing the microfiber fraction will improve insulating capacity and increase materials cost; reducing it will decrease insulating capacity and decrease cost. Our intent to offer a prototype insulator that met the program's thermal conductivity target precluded a decrease in microfiber fraction from the 22/60 value that we selected, but follow-on work should include consideration of an all-macrofiber (1.5 denier P84 and binder) blend. We anticipate that the most significant effect of such a change would be cost reduction. The changes to the insulating performance / cost balance that we foresee are shown in the following table (Table 31):

Table 31. ***Anticipated Effects on Insulating Performance and Materials Cost that Would Result From Elimination of Microfiber from the Prototype P84 Blend***

Blend	Thermal Conductivity (Btu-in/hr-ft^2-°F) at 0.5 lb/ft^3	Thermal Conductivity (W/m-K) at 8.0 kg/m^3	Materials Cost ($/lb)	Materials Cost ($/kg)
22/60/18; micro/macro/binder	0.294	0.042	14.50	31.90
82/18; macro/binder	0.310 Estimate	0.045 Estimate	12.60	27.80

The small increase estimated for thermal conductivity would probably be offset by a small increase in insulator stiffness (resistance to compression), which would mitigate the in-use consequence of the change. Except for, perhaps, a minor increase in the water absorptive capacity of the insulator, no other negative effects of eliminating microfiber from the blend are anticipated.

B. General Functionality and Manufacturing Feasibility

Assessment of the functional role and the overall potential of the newly developed FR insulator can be furthered by comparing its essential characteristics with those of another reference material, Primaloft^{TM}. Primaloft, now a commercial product, originated in a series of research and development efforts, under Natick and Al Research Co. sponsorship, in the 1980s. Its performance characteristics are well known to the Natick staff and, in several cases, provided references for establishing performance targets for this program. Consequently, comparison of the new FR insulator's properties with those of Primaloft (not an FR insulator) and of the needled FR batts of MIL-B-81813B contributes perspective for assessing the new material's merit. Table 32 provides the basis for such a comparison, which leads to the following observations regarding the new FR insulator:

1. It shares virtually all of Primaloft's desirable attributes, i.e., it is down-like in terms of insulating efficiency and compressional characteristics and it has a high level of wetting resistance,

2. In spite of its low density (high loft), it has a high level of flame resistance (as defined by program targets),

3. It is a distinct improvement over Primaloft in terms of laundering durability, as defined by Natick, and

4. It provides a combination of characteristics which neither the MIL-B-81813B battings nor Primaloft offer, being an improvement over each in several ways.

Thus, comparison of the prototype FR insulator's properties with those of the most relevant, state-of--the-art, commercially available insulators provides confidence that it can fulfill broad functional needs. The performance/cost balance of the new insulator, discussed in the previous subsection, strengthens this confidence. Looking ahead, the feasibility of manufacturing the insulator becomes an inevitable topic. Our experience in producing approximately 150 yd^2 of prototype material, reported in Section V, was

Table 32. ***Comparison of the General Characteristics of the FR, WR Insulator Prototype with Those of Three Reference Materials***

Characteristics	Needled Nomex Batting per MIL-B-81813B	Needled Kynol Batting per MIL-B-81813B	Primaloft™	FR, WR Insulator Prototype
Approximate thermal resistance, R (hr-ft^2-°F/Btu) for 4.3 oz/yd^2	0.99	0.99	2.77	2.45
Approximate thermal resistance, (K-m^2/W) for 150 g/m^2	0.17	0.17	0.49	0.43
Volume density (lb/ft^3)[a]	1.4	1.4	0.5	0.5
Volume density (kg/m^3)[a]	22	22	8.0	8.0
Down-like compressional properties	No	No	Yes	Yes
High level of wetting resistance	No	No	Yes	Yes
High level of flame resistance	Yes[b]	Yes[b]	No	Yes
Laundering resistant per "Cotton Procedure" of Method 5556	Yes	Yes	No[c]	Yes

a. Typical use densities. These are the densities upon which the thermal resistance values are based.
b. "Yes" has been assumed. Flammability requirements are not given in MIL-B-818138.
c. Primaloft is very durable in terms of home laundering resistance, but thus far its performance in the "Cotton Procedure" test has been marginal.

unquestionably a positive one and it showed that manufacturing will not be especially difficult. However, brief further discussion of factors influencing manufacturing feasibility is warranted. Several of these factors relate directly to the components of the FR staple blend. These components are:

1. P84 polyimide microfiber; 0.55 denier x 1.5 inch; with fluorocarbon, WR, finish applied by the fiber manufacturer, Lenzing; 22% of blend, by weight.

2. P84 polyimide macrofiber; 1.5 denier x 1.5 inch; with fluorocarbon, WR, finish applied by the fiber manufacturer, Lenzing; 60% of blend.

3. Hoechst Celanese K54 binder fiber; polyester/polyester, sheath/core; 4 denier x 1.5 inch; 18% of blend.

This combination of fiber components contributes several manufacturing advantages to the insulator, as follows:

1. All three components are commercially available.

2. The water repellent finish on both P84 components is applied by the manufacturer, Lenzing (most of the inherently FR fiber candidates considered were not available with producer-applied finish)

3. Carding the blend will be less difficult than carding a blend such as that of Primaloft, which is predominantly microfiber. The larger average fiber diameter of the prototype blend, chosen to slightly compromise insulating efficiency in favor of reduced materials cost, also enhances card-ability.

4. Thermally bonding the heat resistant P84 blend, in comparison to bonding all-polyester, high loft, insulator blends, is less difficult and yields significantly better results. In all of our experience with polyester insulators, the thermal bonding step has been a compromise between loft loss and bonding strength because the polyester matrix softens and collapses, to some degree, within the bonding temperature range. The P84 blend is stable throughout the binder activation range, so that full bond strength can be obtained without great concern for upward temperature drift or overly long dwell time. The laundering durability exhibited by the insulator prototype appears to be the result of optimal bonding.

C. Summary of the Staple-Based, FR Insulator's Potential

From the foregoing, it is evident that the staple-based, FR insulator prototype has near-term potential for fulfilling an important military need. This potential is the result of:

1. The prototype material meeting, with very minor exception, all program performance targets.

2. The prototype exhibiting, in laboratory evaluation, a combination of performance characteristics which neither the FR battings of MIL-B-81813 nor Primaloft offer. The prototype is, in several ways, an improvement over both state-of-the-art insulator types.

3. Reasonable cost.

4. All fiber components being commercially available.

5. Manufacturing feasibility.

6. Versatility; with a minimum of further development effort, the fiber blend ratio can be adjusted to provide a range of performance / cost combinations.

8. VIABILITY OF THE CONTINUOUS FILAMENT, FR INSULATOR CONCEPT

The series of experiences reported in Section 4, *Continuous Filament Insulator Development*, brings into question the viability of that approach to providing a flame-resistant insulator. All who were involved believe that the experiences are representative of what would be encountered should further attempts be made to develop the concept. Several interactive technical problems exist, each requiring empirical solutions based upon difficult, costly, production-line experimentation.

The technical problems that must be resolved can be readily summarized with reference to the final, continuous filament, insulator sample produced in the program. As was reported in Section IV, this sample was deficient in terms of four important performance objectives. Each of these objectives, and the prospects for successfully addressing them, will be discussed separately below:

1. Density. The program density target of 0.3 to 0.6 lb/ft^3 defines a high loft insulator. Previous experience [3,6] has shown that continuous filament, 1.5 denier polyester fiber can be made, with the use of a sprayed resin binder, into a 0.45 lb/ft^3 batt. However, previously used spray resins are not applicable here due to their negative contributions to flame resistance and water repellency. A new binder system will be required to provide the desired high loft, FR, water repellent insulator.

2. Compressional Recovery. Poor fiber opening, non-uniform spreading and batt compaction (high density) were responsible for the poor compressional recovery of the final sample. An appropriate fiber finish, i.e., one that serves the combined needs of opening, spreading, FR, water repellency and bonding is required.

3. Absorptive Capacity. The experience of this and previous work has shown that both silicone and fluorocarbon fiber finishes can impart the desired degree of water repellency to high loft batts made of relatively small-diameter fiber. This experience includes laboratory-applied and production-line-applied finishes of both types. However, Hoechst Celanese has not, to date, been able to apply a silicone finish that is effective as a water repellent. The finish finally adopted must, as noted in 2., above, be compatible with processing and FR needs.

4. Flame Resistance. A contract modification, explained in Section 2, was made to accommodate the relatively poor vertical flammability resistance of Hoechst Celanese' FR polyester. However, none

of the data reported herein indicates that Hoechst Celanese' FR polyester, in a 0.5 lb/ft^3 batt, will meet the revised vertical flammability target. The nonexistence of an alternative, FR, spreadable tow product makes this an issue of primary significance.

Those four performance characteristics, being interdependent, must be considered together. Three of the four, density, compressional recovery and water repellency would be most directly addressed by first developing an acceptable bonding system free from any negative FR effects. Application of a lightweight bonding medium to both batt surfaces immediately after cross-lapping has been shown to be effective. The work of this program included successful laboratory trials with a melt blowing head that deposited a very light, molten, polyester web onto both batt faces. However, extending the technique to production scale will require development, equipment and operating costs that are very high, perhaps prohibitively so. Identification and/or development of a sprayable resin with: (1) improved FR and water repellency characteristics and (2) compatibility with existing spraying and drying equipment appears to be the preferred approach. It will require commitment and development expenditure, but is probably a manageable task. In addition to an acceptable bonding system, an acceptable fiber finish and finish application method must be identified; this is clearly doable.

Although improvements in insulator density, compressional recovery and water repellency can be expected through further work with resin systems and fiber finish, the fourth and final characteristic of concern, flame resistance, is primarily fiber dependent. The relatively poor vertical flammability resistance of Hoechst Celanese FR polyester fiber in lightweight batts, together with the lack of an alternative FR fiber in spreadable tow form, leaves flame resistance as the most problematical performance requirement. Producing tow that can be opened and spread is an art now practiced, with minor exception, only by Hoechst Celanese. Thus, although many inherently FR fiber types were evaluated in the early stages of this work, none is available as spreadable tow and the prospects for any becoming available are not good. Tow is the precursor of all spun and drawn, staple fiber, and, as such, might be made available, but tow not prepared specifically for opening and spreading, with the application of essential proprietary art, will be of no value. Spreadable tow is prepared as a uniform ribbon, rather than as a bundle, has high levels of primary and secondary crimp, and although the ribbon must remain cohesive and intact until fibers are intentionally separated, a lubricating fiber finish is required. The development costs necessary to produce an inherently FR fiber in spreadable tow form are not commensurate with demand; it does not appear likely that Hoechst Celanese or any other fiber producer would undertake such development.

In summary, the effort and costs required to address bonding and water-repellent finish issues would be difficult to justify without the existence of a new, inherently FR continuous filament material. And, as discussed above, cost considerations make development of such a material improbable.

The poor prospects described for the FR, continuous filament, insulator concept should be balanced against the strong potential reported for the FR, staple, insulator approach in preceding sections. Compliance with performance objectives, versatility and the reasonable cost of the staple insulator may foster broader use than originally envisioned and diminish the need for another FR, water-repellent, high performance insulator.

This document reports research undertaken at the U.S. Army Natick Research, Development and Engineering Center and has been assigned No. NATICK/TR-98/013 in the series of reports approved for publication.

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APPENDIX

APPENDIX

Conversion Factors, English System to International System of Units (SI), for Units of Measure Frequently Used in this Report

Thermal Conductivity: $0.144 \ \frac{W}{m\text{-}K} \Big/ \frac{Btu\text{-}in}{hr\text{-}ft^2\text{-}^\circ F}$

Thermal Resistance: $0.176 \ \frac{K\text{-}m^2}{W} \Big/ \frac{hr\text{-}ft^2\text{-}^\circ F}{Btu}$

Volume Density: $16.0 \ \frac{kg}{m^3} \Big/ \frac{lb}{ft^3}$

Areal Density: $33.9 \ \frac{g}{m^2} \Big/ \frac{oz}{yd^2}$

Linear Density: 1.11 dtex/denier

Pressure and Stress: $6.89 \ kPa \Big/ \frac{lb}{in^2}$

Work: 0.113 N-m/lb-in

Mass: 0.454 kg/lb

Length: 2.54 cm/in

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Batting, Aramid or Novoloid Fiber, Quilted, Military Specification MIL-B-81813B

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